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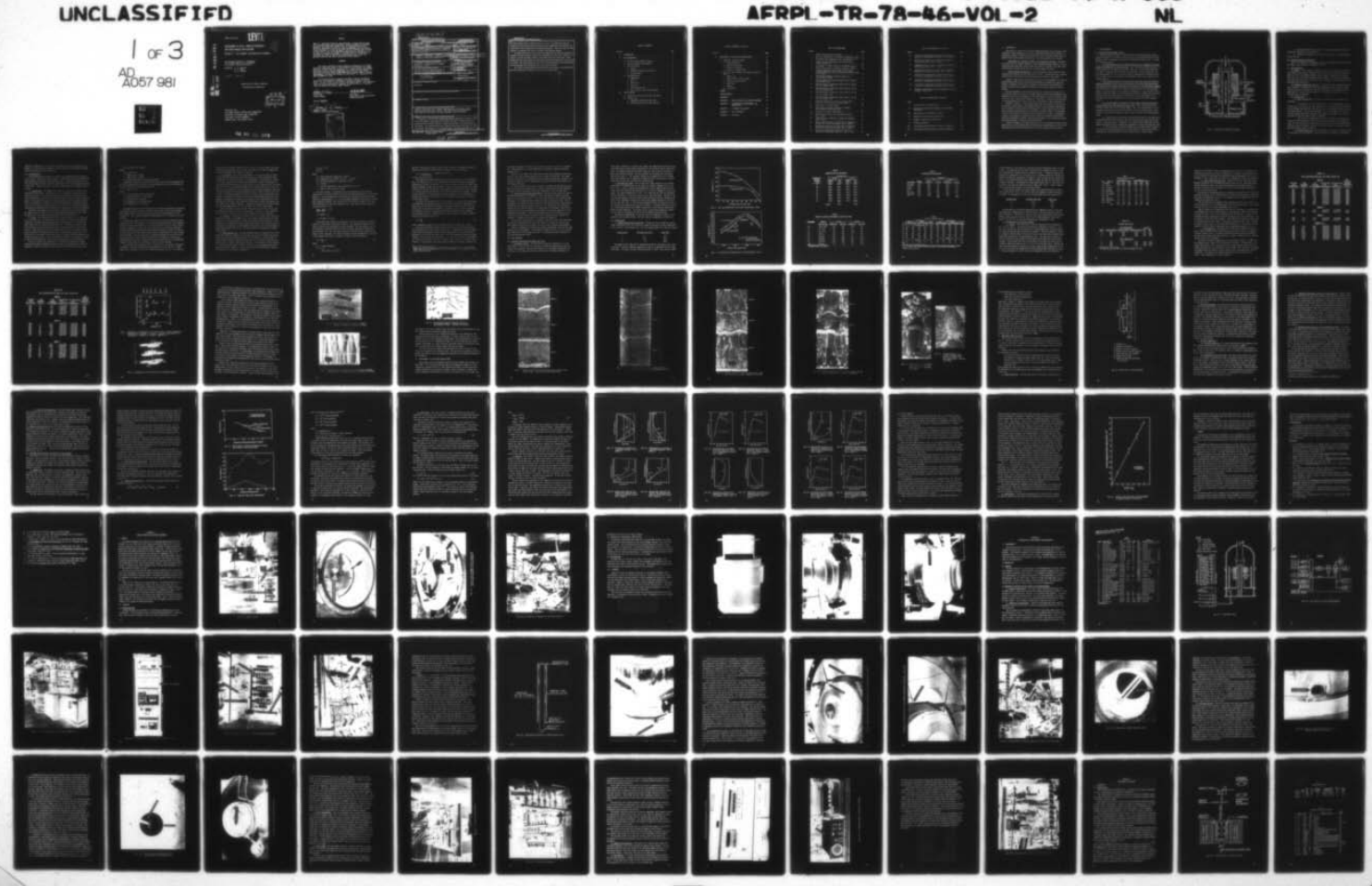

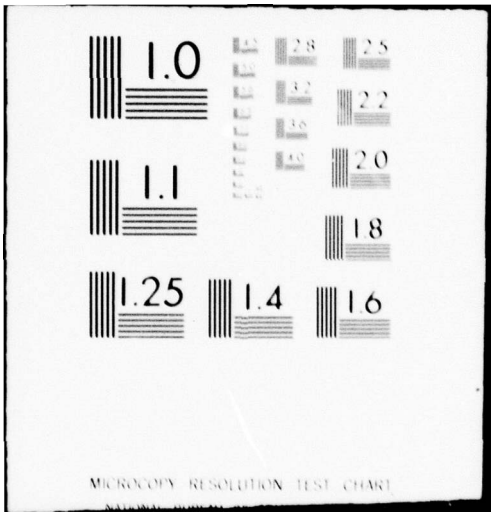
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LEVEL II

DEVELOPMENT OF PG/SiC COMPOSITE MATERIALS FOR ROCKET-NOZZLE APPLICATIONS

VOLUME II - THE CHANNEL FLOW DEPOSITION FURNACE

LOS ALAMOS SCIENTIFIC LABORATORY
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FOREWORD

This report published in three volumes was submitted by Los Alamos Scientific Laboratory (LASL) of the University of California, Los Alamos, New Mexico, 87545, under the auspices of U.S. Energy Research and Development Administration Contract W-7405-ENG. 36, to the Air Force Rocket Propulsion Laboratory (AFRPL) under AFRPL MIPR agreement F04611-76-X-003, JON-305909JQ. The work was directed for LASL by Mr. T.C. Wallace and for AFRPL by Major J.G. Dean.

This report has been reviewed by the AFRPL Technical Information Office and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public. This technical report has been reviewed and is approved for publication; it is unclassified and suitable for general public release.

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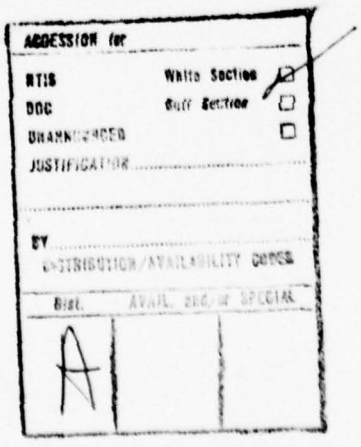

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A series of instrumented engineering tests is described in which a "channel flow" chemical vapor deposition furnace was used to prepare pyrolytic graphite/silicon carbide (PG/SiC) codeposited material for rocket-nozzle applications. The nozzle section and a water cooled center body formed a vertical annular flow passage for the coating gases. The nozzle section was in-

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ductively heated and was coated on the i.d. only. Well-mixed process gas entered the flow annulus with uniform velocity, temperature, and species concentrations from a plenum below the heated substrate. The flow could be either laminar or turbulent, but did not contain recirculating regions.

An analytical model of the furnace and deposition process was developed to simulate the heat transfer in the solid components and the flow, chemical kinetics, and heat and mass transport associated with the process gas. Model predictions and test results on temperatures and PG/SiC deposition rates agreed very well. The coating microstructure and inferred quality were characterized by metallography and correlated with the process variables.

The report includes detailed descriptions of the furnace, instrumentation, and test procedures in addition to plots of all reduced data.

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I. INTRODUCTION

This report is the second of three volumes describing work to develop a more fundamental understanding of the pyrolytic graphite/silicon carbide (PG/SiC) co-deposition process. Hopefully, this work will lead to improved capabilities for making PG/SiC coated rocket-nozzle parts. The overall goals are as follows.

- Development of a analytical model of the deposition process. The analytical model will be complete enough for use in designing furnace geometry and fixtures and specifying power inputs, mass flow rates of reactants, and other input conditions for coating nozzle parts of differing shapes and sizes.

- Control system specification. A fully automatic control system for the deposition process will be specified (see Vol. III). System definition, complete specification, and detailed circuit design and fabrication are not goals.

Volume I[1] summarized the experience that led to the requirement for this program; described the process that had been developed and engineering tests performed to establish a data base for analysis; characterized the deposition kinetics; and described a thermal-flow model of the injector deposition furnace system.

During development of the analytical model for thermal-flow simulation of the injector deposition furnace, it became apparent that existing numerical fluid dynamics codes could not represent the complex separated flow (recirculation flow) in the hot wall configurations adequately. Therefore, a complete analytical model including deposition kinetics could not be developed for that configuration.

Instead, a channel flow deposition furnace was conceived that would be compatible with fluid dynamic code, GENMIX,[2] that had proven modeling capabilities in all requisite areas except recirculating flow. The process gas flow through the new furnace configuration would be simpler (attached, rather than separated, flow), but the deposition kinetics would be essentially the same. Subsequently, a channel flow deposition furnace was built, several instrumented engineering tests were performed to obtain a data base for analysis, and a deposition process model was developed. This work is described here.

Volume III compares the characteristics of the two deposition furnaces, gives some control system specifications, and describes the methodology for use of system modeling as an efficient design tool for PG/SiC coating of nozzle parts.

II. THE EXPERIMENTS

A. Coating furnace design criteria

Using the experience gained with the injector deposition furnace, and because of the need for a furnace configuration that was compatible with the fluid mechanics code, we designed the channel flow deposition furnace, Fig. 1, to meet the following criteria.

1. The design should allow for maximum flexibility in changing the coating fixture and induction coil configurations. This problem was solved by direct induction heating of the nozzle part within a bell jar. This approach minimizes the number of graphite parts that must be fabricated for each coating run, permits rapid changes of coil and coating fixture configurations, increases efficiency of power input to the substrate, and minimizes risks of air leaks into the coating chamber.

2. The process gas should be introduced by some means that will permit accurate specification of the fluid flow parameters at the gas distribution manifold which does not require experimental verification for configurational changes. This criterion was fulfilled by placing a mixing plenum at the coating chamber entrance, and the process gas was injected tangentially to the outer wall from a number of vertical risers in a gas distribution manifold. Making the process gas injection area 100 times larger than the area of the coating chamber, Fig. 1, will make the velocity distribution of the process gas at the inlet constant ($\pm$ 5%).

3. The coating chamber configuration should permit equivalent scaling of flow conditions for throat inserts and nose caps. This criterion was met by placing a center body in the coating chamber. With this configuration, the process gas flow rate will scale as the diameter of the part being coated. Additionally, the center body should be water cooled to provide the following advantages.

a. Radiant heat loss from the substrate being coated to the water-cooled center body will be much greater than the convective heat loss from the substrate to the process gas. Consequently, the axial surface temperature profile along the substrate will be essentially independent of the process gas flow rate.

b. Thin coatings of different emmisivities (polished gold or copper oxide) can be put on the center body in various patterns to give greater design lattitude for achieving constant surface temperatures on complex shapes.

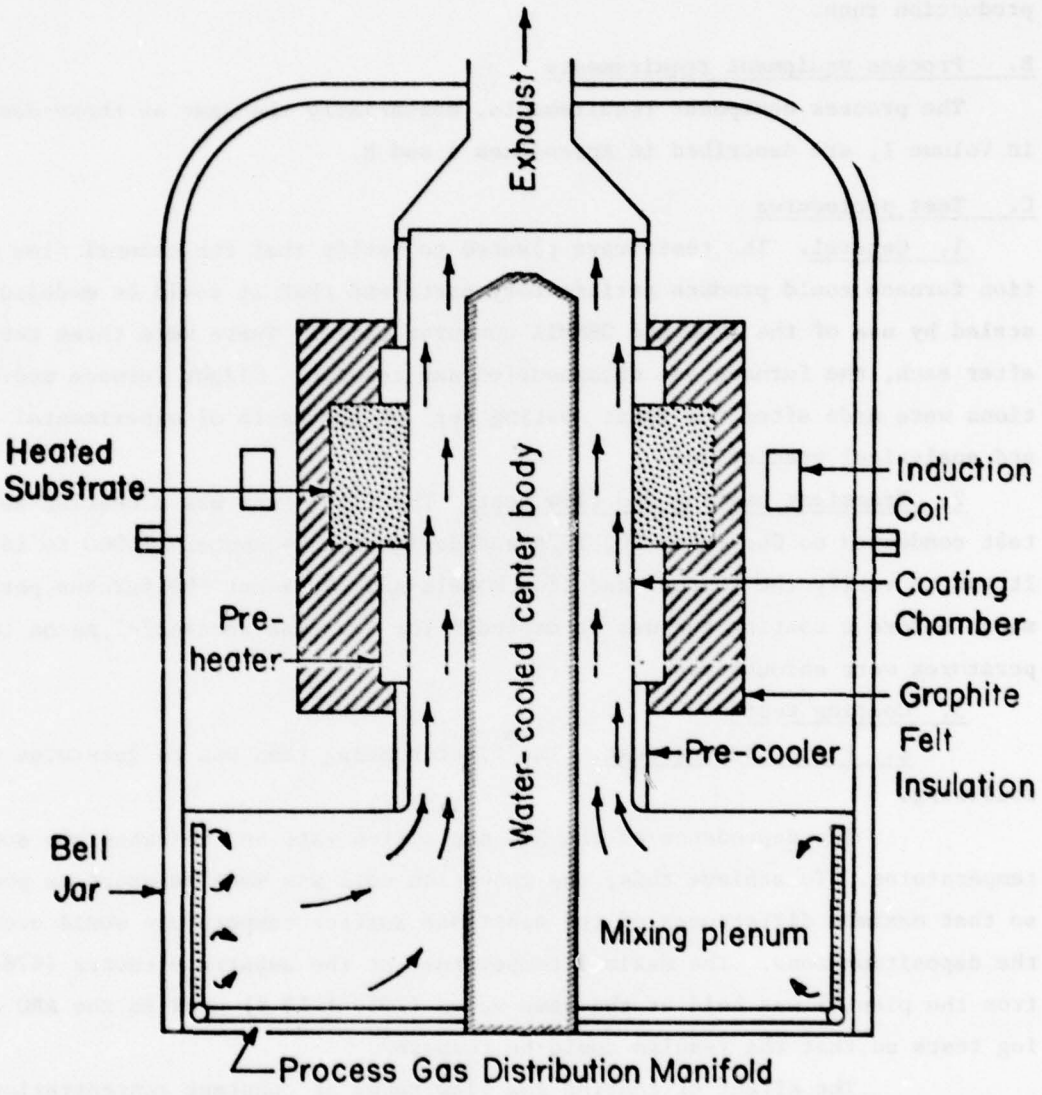


Fig. 1. Channel flow deposition furnace.

c. Coating gas constituents will not be needlessly depleted from process gas by deposition on the center body.

d. Frequent replacement of the center body will not be required during production runs.

B. Process equipment requirements

The process equipment requirements, essentially the same as those described in Volume I, are described in Appendixes A and B.

C. Test procedures

1. General. The tests were planned to verify that the channel flow deposition furnace could produce satisfactory parts and that it could be modeled and scaled by use of the AYER and GENMIX computer codes. There were three tests; after each, the furnace was disassembled and rebuilt. Slight furnace modifications were made after the first coating test on the basis of experimental results and analytical predictions.

2. Transient heating and flow test. The first test was a heating and flow test conducted on September 20, 1976 and designated by numbers 16000 to 16005. It was to verify the thermal and flow models and check-out the furnace performance before a coating run was attempted. The test was successful as no temperatures were encountered.

3. Coating tests

a. First coating test. The first coating test was to determine the following.

The dependence of the SiC deposition rate on the substrate surface temperatures. To achieve this, the induction coil was made as short as possible so that maximum differences of the substrate surface temperature would occur over the deposition zone. The maximum temperature at the substrate center (478 mm from the plenum) was held at the same value (2030 $\pm$ 15 K) used in the ARC coating tests so that the results could be compared.

The effect of coating gas flow rates at constant concentrations on the carbon and SiC deposition rates and on the microstructure of the deposited coat. At the concentration levels used we were able to achieve only 88% (0.091 g/cm^2-s) of the flow used in the ARC tests because we were at the upper range of the Tylan CH_3SiCl_3 (MTS) controller.

b. Second coating test. The objectives of the second coating test were to verify that the SiC deposition rate was essentially constant at 1450-1900 K

substrate temperatures for a given MTS concentration, and to determine the dependence of carbon and SiC deposition rates on CH_4 (1·6 vol%) and MTS (0.16-046 vol%) concentration in the coating gas.

D. Instrumentation

The instrumentation necessary to fulfill the program objectives was determined from the requirements just described. A data acquisition system (DAS) that fully supported the program was designed at LASL. All the equipment was specified and purchased by LASL. Table B-1 (Appendix B) lists the parameters measured in the tests. The more important reasons for specifying these parameters are as follows.

1. Temperatures. The GENMIX code required as input the inside wall temperatures of the furnace. The furnace, Fig. 1, consisted of several parts of different materials. The thermal resistance across a material interface could not be determined accurately, so the heat flow could not be calculated accurately. Hence, the requirement to measure the temperatures of each major part as designated by parameters T-2 through T-9, Fig. B-1 (Appendix B). Thermocouples were used for these temperature measurements (except T-6 and T-7) because the furnace configuration precluded timely installation of sight ports for optical pyrometers.

Additional measurements were necessary to determine the inlet and exhaust gas temperatures (T-1 and T-15) and the room ambient temperature (T-19). The requirements for other temperature measurements are discussed below.

2. Power. The power generated in the furnace susceptor is a necessary input to the AYER code. Determining the power required 7 cooling water flow measurements, 10 temperature measurements, and the measurements to calculate the power supplied by the 10-kHz motor-generator set. The susceptor power is that which remains from the motor-generator output after the losses have been subtracted. These losses include the resistive (I^2R) losses of the induction coil, and the shunting and resistive losses in the capacitor bank and bus bar. The induction coil was paralleled with capacitance to present the motor-generator set a power factor near unity. This occurs when the current and voltage are in phase so that the motor-generator can deliver its rated power at rated efficiency.

The total furnace power parameter W-1 (EI COS O) was measured by signal conditioning equipment designed and built at LASL. The parameter W-2 (E-1) was measured to verify the electrical power factor to which the furnace was tuned.

$$PF = EI \cos \phi / EI = \cos \phi \quad (1)$$

where

PF = power factor

E = RMS value of voltage

I = RMS value of current

ϕ = phase angle between voltage and current

Assuming an adiabatic process, the I^2R loss in the coil was determined from Eq. (2) by measuring the flow rate (F-8) and temperature of the cooling water at the coil inlet (T-11) and discharge (T-18).

$$Q = mCp\ (T_2 - T_1), \quad (2)$$

where

Q = power removed by the cooling water

m = mass flow rate of cooling water

C_p = specific heat (constant)

T_2 = temperature of discharge water

T_1 = temperature of inlet water

The power removed by the remaining furnace fixtures was similarly calculated by measuring the water flow rate in these circuits and the inlet and discharge water temperatures. The seven cooling water flow rate measurements are F-6 through F-12. The 10 water temperature measurements are T-11 through T-18, T-20, and T-21.

Only three flowmeters were used to measure the flow rate through the seven cooling circuits. The flow rate of six circuits was measured with two flowmeters (three flow rates per flowmeter). By valving the water of one of three circuits through a flowmeter, one at a time, the flow rates of all three were measured. To ensure that the system pressure would remain reasonably constant, all but two of the cooling water loops were flowing during the switching of circuits through the flow meters. The third flow meter constantly measured the flow rate of one loop only to pick up any pressure fluctuation in the supply as circuits were being switched in and out. Pressure fluctuations would show up as corresponding changes in flow rates that could then be used to correct the data.

The radiation and conduction losses to the bell jar, and then to the room's environment, required the temperature measurements T-5, T-6, and T-19. These supplied the temperature data for the simplified equation $Q_T = h_r + h_c$, where Q_T

equals the total energy transferred and h_r and h_c are characteristic functions of the furnace materials, the bell jar, the furnace room, and the temperature differences between their respective surfaces.

3. Flow Rates. An accurate measurement of the reactant gas flow rates (along with some temperature data) was necessary to obtain information about the kinetics of the PG/SiC process. The measurement of nitrogen (F-1) and MTS (F-2) flows along with the inside wall temperatures, helped to determine and verify the MTS disassociation rate with respect to the temperature and the deposition rate of SiC. An accurate determination of the mass flow rate of CH_4 (F-3 and F-13), was required to help determine and verify which parameters controlled (or were a function of) the total deposition rate of PG/SiC and the ratio of PG to SiC. An accurate measurement of these flow rates and wall temperatures was necessary to establish a data base for furnace scaling requirements and to determine, if possible, the reason for the previous poor reproducibility of the ARC deposition process. Flow controllers were used to adjust and regulate the flow rates of CH_4 and MTS reactants. Inherently the deposition process requires accurate measurement and control of the mass flow rates of these gases.

Small fluctuations in the nitrogen flow were not critical to the process, but knowing the actual flow rate was necessary. Therefore, a flow controller was not used for this gas, but a accurate flowmeter (F-1) was installed.

4. Pressure. The absolute pressure (P-1) at the furnace inlet plenum was measured for input to the GENMIX code. The pressure along with the inlet temperature (T-1), was required to determine the density of the inlet gas. These parameters, in conjunction with exit temperature (T-10), and nitrogen flow (F-1) satisfied the requirement for determining the energy picked up by the gasses as they passed through the furnace. This was required to determined the furnace energy balance.

5. Data Sampling Rate. The data sampling rates were specified after estimating the transient response of the furnace and the PG/SiC deposition process. After reviewing the physical properties of the furnace, it was felt that its characteristic equation expressing temperature as a function of time would be a second order (or higher) differential equation. The transient furnace response to a step power input would be similar to that of an over-damped second-order electrical system. However, a "worst case" solution was obtained by modeling the furnace as a first-order system (Eq. 3).

$$\frac{T_f - T_t}{T_f - T_i} = e^{\frac{-t}{\tau}} \qquad (3)$$

where

T_f = Final substrate temperature = 2033 K

T_t = Temperature of substrate at time t = 2028 K

T_i = Initial temperature of substrate = 294 K

e = 2.71828

t = time for substrate to reach temperature T_t = 3 h

τ = Furnace time constant in hours

The time constant τ is defined as the time required for the output to reach 63.2% of its final steady-state value after being subjected to a step input function. It was estimated that the substrate would achieve steady state temperature in approximately 3 h. If the transient response of the furnace followed this simplified equation, the furnace would reach 99.75% or within 5 K of its final temperature in six time constants (3 h). This furnace time constant was calculated from Eq. (3),

$$\frac{2033 - 2028}{2033 - 294} = e^{\frac{-3}{\tau}}$$

$$\ell n\ 0.00288 = -\frac{3}{\tau}$$

$$\tau = 0.51282 \text{ h} = 31 \text{ min.}$$

The furnace time constant is analogous to that of an electrical system defined by a first-order differential equation. The frequency response of this type of system can be obtained from a Bode diagram.[3] On this diagram, the corner frequency is defined as that at which the output amplitude of the system has decreased 3 db. The 3-db system frequency response is then bounded by zero and the corner frequency. The corner frequency is also identified as a function of the system time constant. Equation (4) depicts this correlation.

$$f_{cf} = \frac{1}{2\P\ \tau}\ , \qquad (4)$$

where

f_{cf} = corner frequency

$\P$ = 3.14

τ = time constant of system

By use of this equation and the furnace time constant, the highest frequency to which the furnace will respond (corner frequency) can be calculated.

$$f_{cf} = \frac{1}{(2)(3.14)(31)} = 0.0051 \text{ cycle per min}$$

or one cycle per 195 min.

The sampling theorem stipulates that if the rms spectrum of a time function g(t), is identically zero at all frequencies above W Hz, then g(t) is uniquely determined by giving its ordinates at a series of points spaced 1/2 W apart, the series extending through the time domain. However, for this theorem to be valid, a perfect filter must be applied to the signal of interest with a cutoff at W Hz, or a perfect signal spectrum with no energy above W Hz must be sampled. Neither case is practical, so the sampling rate must be greater than 2 W samples/s to prevent the aliasing* error, present in all sampling data systems, from being exceptionally large.

To reduce the aliasing error[4] to less than 1% in a time division multiplexing (TDM) data system with two poles of filtering, the ratio of sampling frequency (f_s) to the signal 3db frequency (f_{cf}) must be less than 30. Here the two poles of filtering are provided by the furnace, as its transient response is defined by at least a second-order differential equation with a damping ratio greater than one. Substituting the corner frequency of 0.0051 cycle/min which was calculated above, into

$$30 = \frac{f_s}{f_{cf}} \tag{5}$$

gives a sampling frequency of 0.153 samples/min or one sample per 6.5 min.

The process variables, power, reactant flow rate, and cooling water flow rate had much faster time constants (about 20 s). However, the furnace temperatures could not respond this fast, and the resolution of the process was not definable to this relatively short time. Experience showed that after a drastic change in one of the process variables, (i. e., MTS flow turned off) approximately 15 min was required to observe a change in the process. If 15 min is two-thirds of a time constant, the process time constant is 22.5 min. From Eq. (4)

*Simply the misrepresentation of the frequency and amplitude of the recorded signal when the data-sampling rate is too low relative to the frequency of the signal being measured.

the fequency response is then one cycle per 141.3 min or a rate of 4.7 min/sample. The frequency of the room temperature fluctuations required a similar sampling rate.

Therefore, the sample rate used during steady state operation was one sample per 5 min, which seemed adequate from the above calculations. The sample rate was increased to one sample/min during transient heating and cooling of the furnace to insure no loss of information during these periods from unknown furnace characteristics.

It is not certain that the error because of aliasing in TDM systems is germane to this particular application. However, ensuring that the data-sampling rate meets these requirements lends more credibility to the recorded data.

6. Data Accuracy and Recording Method. The end-to-end inaccuracy requirements were specified as $\pm$ 2% maximum. This requirement was based on several factors, one being the accuracy of data required as inputs to the AYER and GENMIX codes or for comparison with conditions predicted by these codes. The requirements to ascertain the process kinetics and reproducibility characteristics dictated an inaccuracy no greater than $\pm$ 2%. The parameters controlling the process were not entirely known at the beginning of the program. Therefore, to relate the effect of one parameter upon another, an accurate signature of each parameter was necessary. The $\pm$ 2% requirement was also a prerequisite for an accurate data base on which a process control system was to be designed and specified. Instrument calibration is discussed in Appendix C.

The data were recorded on $\frac{1}{2}$ - in magnetic tape in a seven-track IBM format. The data-recording medium and format were chosen to allow easy access to the LASL computer facility and to reduce the cost of data reduction and handling.

Table B-1 (Appendix B) outlines the complete measurement list and the range required for each parameter. All parameters except F-2, F-3, F-4, F-5, and W-2 were required for the nitrogen flow test. All measurements were required for the coating deposition tests.

III. DATA OBTAINED

A. Transient heating and nitrogen flow tests

1. Transient heating. The transient heating test consisted of a step power input with zero gas flow, starting from existing temperatures after the furnace was outgassed. The data were taken for the transient heating part of this test in case unexpected problems developed in the thermal modeling. It

would then be possible to calculate and compare the temperatures during the heat-up starting with a known uniform temperature in the furnace, without significant flow. No unexpected problems developed, (App. D), and it was not necessary to model the transient heating. The data obtained are included with the reduced data plots in App. E. The transient heating is an integral part of the normal start-up procedure, and no significant additional effort was required to obtain these data for each run (including the coating tests).

2. Nitrogen flow tests. The nitrogen flow tests consisted of two steady-state holds at constant power and two levels of constant nitrogen flow (11 ℓ/s and 19 ℓ/s). After it had been determined that furnace temperatures had stabilized, each test condition was maintained for 1 h with no changes in control settings. The radial temperatures of the ∿ 3 in. diam exit gas tube were measured at each flow level to verify the correction for stem conduction. The gas in the exit tube was assumed to be sufficiently mixed and nearly uniform temperature because of the turbulence caused by the abrupt area changes between the flow annulus and exit tube, Fig. 1. The exit gas temperature data are presented in Fig. 2. The corrected gas temperatures are given in Table I. Standard methods and literature values were used for the thermal conductivity of the tantalum thermocouple sheath and BeO insulation. The change in the corrected gas temperature between the two flows is consistent with the expected change from gas heating.

The measured temperatures for the two flows are summarized in Table II and compared with calculated values. The heat balance is summarized in Table III.

B. Coating runs.

1. Temperatures during coating runs. During the first coating run, Series 17000, the furnace power was controlled to keep the substrate surface temperature, (T-7) at 2020 K. The nitrogen flow, (F-1) and furnace power (W-1) were as follows:

Coating Layer	Nitrogen Flow (ℓ/s)	Power (kW)
1	14.3	82.0
2	11.0	82.0
3	7.8	89.0

The range of flow rates had no significant effect on the furnace power level to maintain 2 constant substrate temperature This is in agreement with the model predictions. The power increase during deposition of layer 3 was probably caused

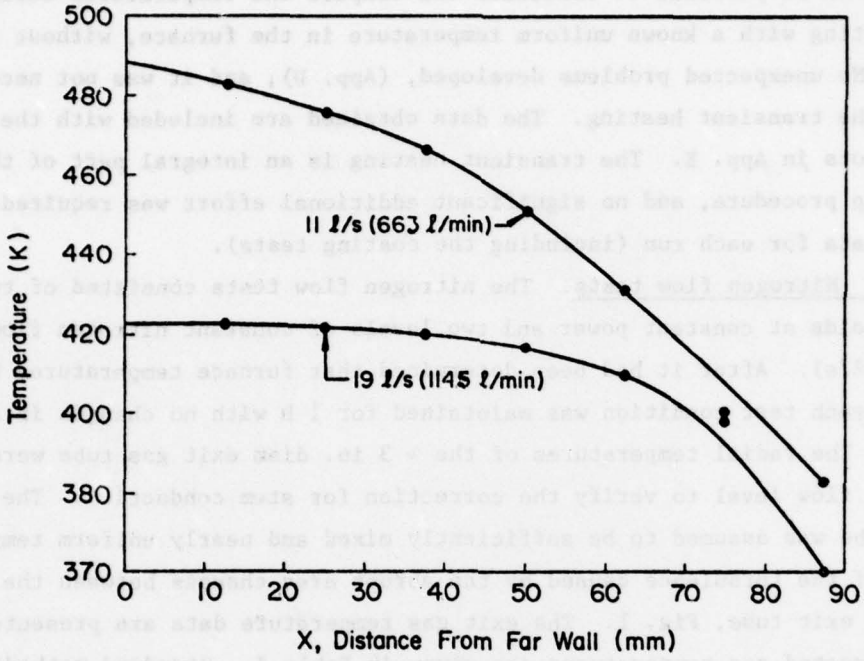


Fig. 2. Exit gas temperature from radial thermocouple scans.

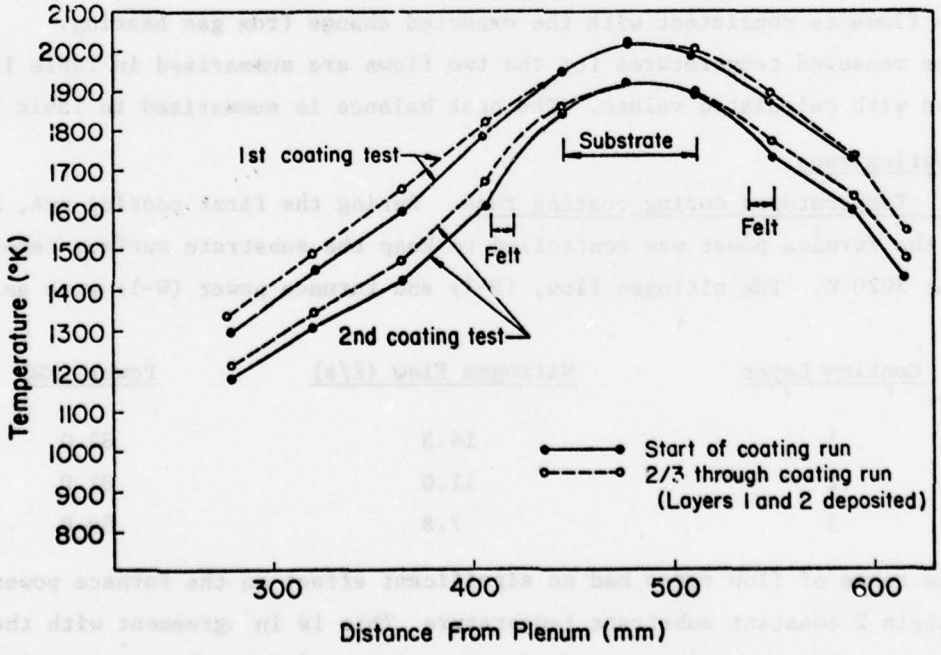


Fig. 3. Calculated wall temperatures for Coating Runs 1 and 2.

TABLE I

CORRECTED EXIT GAS TEMPERATURES

Thermocouple Position from wall (mm)	Nitrogen Flow 11 ℓ/s Measured (K)	11 ℓ/s Gas (K)	19 ℓ/s Measured (K)	19 ℓ/s Gas (K)
13	483.0	501.1	422.7	426.2
25	476.0	501.1	421.7	425.7
38	466.6	501.0	420.3	425.5
50	450.9	501.9	417.3	425.7
64	431.4	501.9	409.9	426.4
76	400.5	502.5	391.9	427.7
89	382.9	500.0	370.0	428.2
	Average	501.4		426.5

TABLE II

MEASURED AND CALCULATED TEMPERATURES IN NITROGEN FLOW TESTS

Measurement	Location (See Fig. B-1)	11 ℓ/s Measured (K)	11 ℓ/s Calc. (K)	19 ℓ/s Measured (K)	19 ℓ/s Calc. (K)
T-2	Bottom, inlet	1593	1460	1558	1501
T-3	Mid, inlet	1762	1594	1712	1610
T-4	Top, inlet	1790	1706	1629[a]	1788
T-5	Felt OD	515	626	511	644
T-6	Susceptor	2204	2138	2173	2218
T-7	Substrate	2033	2044	2090	2088
T-9	Exit wall	2135	1850	2166	1878
T-10	Gas exit	501	571	427	455

[a] Probably in error. See App. B.

TABLE III

HEAT BALANCE FOR NITROGEN FLOW TESTS

Cooling Circuit	Water Flow (g/s)	Nitrogen Flow 11 g/s Exit Temperature (K)	11 g/s Power (kW)	19 g/s Exit Temperature (K)	19 g/s Power (kW)
Upper Canopy	0.236	309.3	7.1	309.8	6.5
Coil Support	0.345	302.4	0.40	303.4	0.32
Bell Jar	3.59	303.1	14.1	304.0	11.7
Precooler	0.251	307.0	5.1	307.8	4.8
Coil	0.349	318.1	23.3	319.3	23.5
Center body	0.494	311.7	19.8	313.4	21.1
Capacitor Bank	0.194	307.6	4.5	308.6	4.4
Bus Bar	0.208	302.83	0.62	303.9	0.59
			76.5		74.3

TABLE IV

MEASURED AND CALCULATED TEMPERATURES

T	1st Coating Run Layer 1 Measured	Layer 1 Calculated[a]	Layer 2 Measured	Layer 2 Calculated[a]	Layer 3 Measured	2nd Coating Run Layer 1 Measured	Layer 1 Calculated[a]	Layer 2 Measured	Layer 2 Calculated[a]	Layer 3 Measured
T-2	----------Instrument Failed----------					1451	1278	1493	1312	1549
T-3	1414[b]	1553	1449[b]	1598	1468[b]	1311[b]	1387	1343[b]	1430	1397[b]
T-4	2010	1669	2109	1712	2122	1482	1486	1525	1536	1583
T-5	645	704	669	762	685	443	635	459	684	478
T-6	2397	2186	2490	2219	2504	2221	2079	2396	2102	2460
T-7	2021	2020	2015	2021	2019	1914	1922	1920	1914	1917
T-8	1775	1840	1818	1879	1846	----------Missing----------				
T-9	----------Missing----------					1600	1692	1627	1722	1672
T-10 (Gas)	447	----	486	----	537	576	----	570	----	533

[a] The first column of calculated data corresponds to the start of the test with no coating present. The second column of calculated data corresponds to the point 2/3 through the test with layers 1 and 2 present.

[b] Probably in error, see App. E.

by deterioration of the carbon felt insulation and the insulating effect of layers 1 and 2. The measured and calculated temperatures are compared in Table IV, and the calculated wall temperatures are presented in Fig. 3. Measured temperatures upstream and downstream of the substrate tend to increase during the coating runs. whereas the substrate temperature is constant. This is caused by the insulating effect of the first two layers which tends to direct the heat away from where the coating is thickest. This agrees with model predictions, Fig. 3. The effect was included in the flow and chemical kinetics models (Sec. IV.B.). Table V summarizes the water heat pickup in the various circuits.

For the second coating run, Series 18000, the furnace and model were modified slightly (Sec. IV.A). The furnace power was adjusted to keep the substrate at 1920 K. The nitrogen flow, (F-1) and furnace power (W-1) are listed below.

Coating Layer	Nitrogen Flow (ℓ/s)	Power (kW)
1	4.74	80.6
2	4.74	81.3
3	7.9	93.0

The measured and calculated temperatures are compared in Table V, the coating chamber wall temperatures are given in Fig. 3 , and the water heat pickup is shown in Table VI. The ratio of the power lost to the center body in the corresponding coating layers of the two tests is 0.8, which agrees with the fourth power of the ratio of the substrate temperatures, $(1920/2020)^4 = 0.82$.

2. Characterization of PG/SiC deposits.

a. General. The objectives of the two coating runs are presented in Sec. II.C.3. a and b. The process conditions are summarized in Table VI. For the first coating run, the flow rates of all process gases were reduced uniformly to keep the CH_4 and MTS concentrations constant. This was expected to affect the coating's microstructure (from good to poor as the flow rate decreased) while maintaining essentially the same deposition rate. For the second coating run, the CH_4 concentration was varied for each layer and the MTS flow rate was held constant. The nitrogen flow rate was the same for the first two layers and increased to agree with that of layer 3 of run 1 for the final layer. The MTS concentration was therefore constant for the two levels of CH_4 concentration in the first two layers and was reduced while the CH_4 concentration increased for layer 3. This was expected to verify that the SiC deposition rate was essentially

TABLE V

SUMMARY OF WATER HEAT PICKUP (kW)

T_{out}	Location	1st Coating Run			2nd Coating Run		
		Layer 1	Layer 2	Layer 3	Layer 1	Layer 2	Layer 3
	Test Series	17005	17007	17009	18004	18006	18008
T-12	Bell Jar	10.3	11.9	11.8	9.3	11.9	12.7
T-13	Coil Support	0.5	0.6	0.5	0.4	0.6	0.7
T-14	Centerbody	28.7	32.2	33.7	22.3	25.7	24.9
T-15	Bell Jar Base	2.0	2.2	2.0	1.1	1.3	1.3
T-16	Lower Precooler	3.1	3.4	3.4	2.9	3.2	3.3
T-17	Coil	27.4	35.5	39.4	31.3	43.1	49.5
T-18	Upper Canopy	6.0	6.0	5.9	5.1	5.1	5.1
T-20	Capacitor Bank	4.3	4.8	5.0	4.6	5.4	6.0
T-21	Bus Bar	0.7	0.8	0.7	0.6	0.8	0.9
	Total	83.0	97.4	102.4	77.6	97.1	104.4

TABLE VI

PROCESS CONDITIONS

Layer	N_2(std ℓ/min)[a]	CH_4(std ℓ/min)[a]	MTS(std ℓ/min)[a]	Vol % CH_4	Vol % MTS
		1st Coating Run			
1	850	8.59	1.38	1.0	0.16
2	661	6.67	1.07	1.0	0.16
3	472	4.67	0.76	1.0	0.16
		2nd Coating Run			
1	283	18.0	1.34	5.97	0.46
2	283	9.0	1.34	3.07	0.46
3	472	23.0	1.34	4.63	0.27

[a]To convert from std ℓ/min to g/min, multiply by 6.67.

constant for 1400-1900 K substrate temperatures and to verify the dependence of carbon and SiC deposition rates on concentration. Further, it was expected that the two deposition rates would be essentially mutually independent (SiC rate independent of CH_4 concentration and carbon rate independent of MTS concentration).

b. Coat characterization data. Tables VII and VIII summarize experimentally determined coat thickness and weight percent of SiC, calculated carbon and SiC deposition rates, and calculated substrate surface temperatures as a function of distance from the plenum for each of the three layers deposited in the 1st and 2nd coating runs.

Procedures for determining the coat thickness and weight percent of SiC, estimating the density of the coats from the SiC content, and calculating the deposition rates are described in Vol. I.[1] The substrate surface temperatures are calculated values taken from Fig. 3.

The data show that the maximum carbon deposition rate for each layer is directly proportional to the volume percent of CH_4 in the process gas. This correlation was verified in the model development (Sec. IV.B).

Figure 4 shows the SiC deposition rate data as a function of reciprocal surface temperatures for the layers deposited. Unfortunately, there is more scatter in these data than in those for the carbon Tables VII and VIII. However, for a specified initial concentration of MTS in the process gas, the SiC deposition rate is constant ($\pm$ 15%) from 1400 to 1900 K. Above 1900 K, the deposition rate drops off, approaching zero near 2100 K. The absolute deposition rate (1400 - 1900 K) is directly proportional to the initial MTS concentration. This behavior agrees generally with the injector deposition furnace experience discussed in Vol. I.

c. Microstructure. The properties of the PG/SiC codeposited material may be viewed as being derived from the basic properties of PG by the addition of accicular crystal of SiC. To better understand the microstructures developed, we will start by discussing the PG structure. Figure 5 shows the atomic arrangement of a unit cell of graphite.

The distance between carbon atoms in the A-B plane is smaller (1.42Å) than that between carbon atoms (3.97Å) in adjacent A-B planes. Correspondingly, the carbon-carbon bonds in the A-B plane are very strong, whereas those between adjacent A-B planes (in the C-direction) are very weak. This directional difference in bonding within the graphite lattice leads to very large anisotropic property variations. For example, the thermal conductivity in the C-direction is $\sim$ 1/100 that perpendicular to the C-direction.

TABLE VII

COAT CHARACTERIZATION DATA FOR FIRST COATING RUN

Distance from Plenum (mm)	Coat Thickness (mils)	SiC Content of Coat (wt%)	C Deposition Rate (moles/cm^2-s)	SiC Deposition Rate (moles/cm^2-s)	Mean Substrate Surface Temperature (K)
Layer 1					
332	4.0	28.8	$1.13 \cdot 10^{-7}$	$1.37 \cdot 10^{-8}$	1485
357	6.6	22.7	$1.99 \cdot 10^{-7}$	$1.75 \cdot 10^{-8}$	1570
386	9.2	16.7	$2.93 \cdot 10^{-7}$	$1.76 \cdot 10^{-8}$	1695
408	12.3	12.6	$4.06 \cdot 10^{-7}$	$1.75 \cdot 10^{-8}$	1795
438	12.8	5.3	$4.52 \cdot 10^{-7}$	$0.76 \cdot 10^{-8}$	1920
462	11.8	3.9	$4.21 \cdot 10^{-7}$	$0.51 \cdot 10^{-8}$	1990
487	10.0	3.0	$3.60 \cdot 10^{-7}$	$0.33 \cdot 10^{-8}$	2020
512	7.8	4.5	$2.77 \cdot 10^{-7}$	$0.39 \cdot 10^{-8}$	1990
550	5.8	10.4	$1.97 \cdot 10^{-7}$	$0.68 \cdot 10^{-8}$	1870
575	5.2	14.0	$1.70 \cdot 10^{-7}$	$0.83 \cdot 10^{-8}$	1780
Layer 2					
386	12.2	13.8	$3.54 \cdot 10^{-7}$	$1.67 \cdot 10^{-8}$	1715
408	14.1	12.6	$4.09 \cdot 10^{-7}$	$1.77 \cdot 10^{-8}$	1815
Layer 3					
386	6.4	12.0	$3.72 \cdot 10^{-7}$	$1.52 \cdot 10^{-8}$	1735
408	7.0	9.7	$4.16 \cdot 10^{-7}$	$1.34 \cdot 10^{-8}$	1831
Layers 2 + 3					
332	6.5	36.9	$0.98 \cdot 10^{-7}$	$1.72 \cdot 10^{-8}$	1535
357	10.0	21.5	$1.78 \cdot 10^{-7}$	$1.46 \cdot 10^{-8}$	1620
408	21.1	9.4	$4.19 \cdot 10^{-7}$	$1.30 \cdot 10^{-8}$	1830
438	21.1	4.6	$4.38 \cdot 10^{-7}$	$0.63 \cdot 10^{-8}$	1920
462	19.6	2.2	$4.15 \cdot 10^{-7}$	$0.28 \cdot 10^{-8}$	2000
487	17.0	2.0	$3.60 \cdot 10^{-7}$	$0.22 \cdot 10^{-8}$	2030
512	13.8	2.6	$2.91 \cdot 10^{-7}$	$0.23 \cdot 10^{-8}$	2000
550	10.5	10.1	$2.07 \cdot 10^{-7}$	$0.70 \cdot 10^{-8}$	1895
575	8.7	11.2	$1.70 \cdot 10^{-7}$	$0.64 \cdot 10^{-8}$	1800

TABLE VIII

COAT CHARACTERIZATION DATA FOR SECOND COATING RUN

Distance from Plenum (mm)	Coat Thickness (mils)	SiC Content of Coat (wt%)	C Deposition Rate (moles/cm^2-s)	SiC Deposition Rate (moles/cm^2-s)	Mean Substrate Surface Temperature (K)
			Layer 1		
438	70	6.8	$2.13 \cdot 10^{-6}$	$4.66 \cdot 10^{-8}$	1815
462	73	5.7	$2.24 \cdot 10^{-6}$	$4.06 \cdot 10^{-8}$	1895
487	66	6.1	$2.01 \cdot 10^{-6}$	$3.91 \cdot 10^{-8}$	1920
512	53	5.6	$1.63 \cdot 10^{-6}$	$2.99 \cdot 10^{-8}$	1885
550	23	16.9	$0.64 \cdot 10^{-6}$	$3.99 \cdot 10^{-8}$	1730
575	16	22.5	$0.42 \cdot 10^{-6}$	$3.67 \cdot 10^{-8}$	1650
			Layer 2		
360	5	51.1	$0.09 \cdot 10^{-6}$	$2.92 \cdot 10^{-8}$	1435
386	9	43.6	$0.19 \cdot 10^{-6}$	$3.85 \cdot 10^{-8}$	1540
438	36	13.1	$1.05 \cdot 10^{-6}$	$4.72 \cdot 10^{-8}$	1830
462	37	9.5	$1.10 \cdot 10^{-6}$	$3.46 \cdot 10^{-8}$	1900
487	33	9.6	$0.97 \cdot 10^{-6}$	$3.07 \cdot 10^{-8}$	1920
512	25	12.9	$0.71 \cdot 10^{-6}$	$3.14 \cdot 10^{-8}$	1890
550	16	22.9	$0.41 \cdot 10^{-6}$	$3.62 \cdot 10^{-8}$	1750
575	11	31.0	$0.27 \cdot 10^{-6}$	$3.57 \cdot 10^{-8}$	1685
			Layer 3		
360	4	32.5	$0.10 \cdot 10^{-6}$	$1.46 \cdot 10^{-8}$	1460
386	5	30.7	$0.13 \cdot 10^{-6}$	$1.71 \cdot 10^{-8}$	1560
408	12	12.7	$0.37 \cdot 10^{-6}$	$1.62 \cdot 10^{-8}$	1685
438	47	2.9	$1.59 \cdot 10^{-6}$	$1.42 \cdot 10^{-8}$	1845
462	48	3.5	$1.62 \cdot 10^{-6}$	$1.76 \cdot 10^{-8}$	1905
487	41	4.1	$1.35 \cdot 10^{-6}$	$1.73 \cdot 10^{-8}$	1920
512	29	4.2	$0.96 \cdot 10^{-6}$	$1.27 \cdot 10^{-8}$	1895
550	19	7.8	$0.60 \cdot 10^{-6}$	$1.51 \cdot 10^{-8}$	1770
575	13	13.7	$0.43 \cdot 10^{-6}$	$2.02 \cdot 10^{-8}$	1720

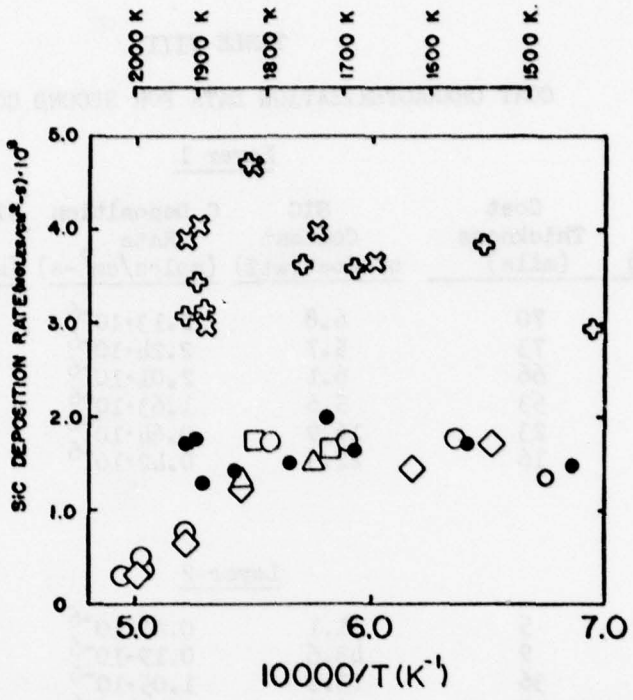


Fig. 4. Variation of SiC deposition rate with recipocal surface temperature. Coating Run 1: ○-Layer 1, □-Layer 2, △-Layer 3, ◇-Layer 2 + 3. Coating Run 2: ✡-Layer 1, ✧-Layer 2, ●-Layer 3.

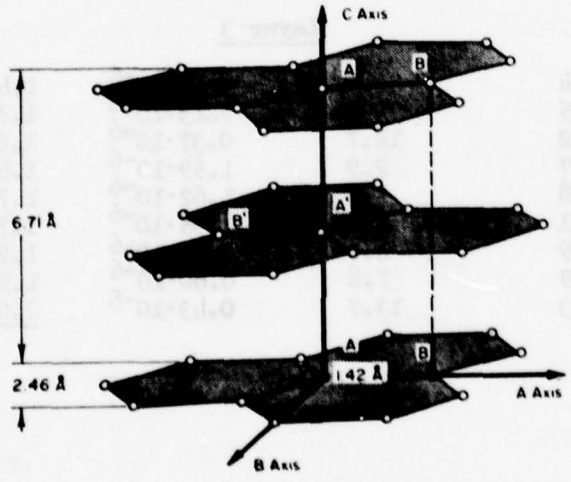


Fig. 5. Arrangement of carbon atoms in the graphite lattice.

The anisotropic properties also appear in samples that have been hydrogen ion etched before scanning electron microscope (SEM) examination. Figure 6 is a (SEM) photomicrograph of substrate graphite that shows differential etching characteristics in the C-direction (fish scales -- one is looking at the A-B plane) and perpendicular to the C-direction (lammellae).

In the chemical vapor deposition process, SiC is codeposited with PG as accicular crystals. The A-B plane of the PG is approximately parallel to the graphite substrate surface, whereas the SiC needles are perpendicular to the deposition surface (and the A-B planes of the PG). During deposition, the PG does not grow as a single crystal (all A-B planes exactly parallel to the deposition surface) but as a collection of crystallites that are somewhat disarrayed with respect to one another. The individual crystallites assemble into polygonal zones or grains connected by tilt-boundaries (cone-boundaries) and look like wrinkled sheets. Figure 7 is a photomicrograph of the three layers deposited in Coating Run 1 (386 mm from the plenum) which illustrates these characteristices. The spatial orientation of the lattices of adjacent grains (separated by a cone-boundary) will be different, so a SEM of the hydrogen-ion etched area will show different structures.

Figure 8 is a SEM photomicrograph of adjacent grains. The area on the left shows the fish scale appearance of the A-B plane of graphite and the needle-like SiC crystals appear to be protruding through the A-B plane. The area on the right has the lamella appearance characteristic of an orientation perpendicular to the C-direction and the SiC crystals appear to be lying on the surface.

Figures 9 and 10 are photomicrographs of the deposited layers from coating run 1, 386 and 408 mm from the plenum. In proceeding from layer 1 to layer 3, the process gas flow rate was decreased, while the volume percent of CH_4 and MTS was held constant (Table VII). Correspondingly, the microstructures change from a uniform distribution of accicular SiC within the grains (layer 1) to coarse SiC crystals concentrated in the cone-boundaries (layer 3). The sequence of microstructural changes with decreasing flow rate is also observed in the injector deposition furnace.[1]

Figures 11-14 show the microstructure of the layers deposited in run 2. Layers 1 and 2 were deposited at lower flow rates than were used for the corresponding layers in run 1, whereas the flow rates for the respective layers were the same. As expected, the microstructures of the layers in run 2 were not characteristic of good codeposited material. Figure 15 shows the development of an

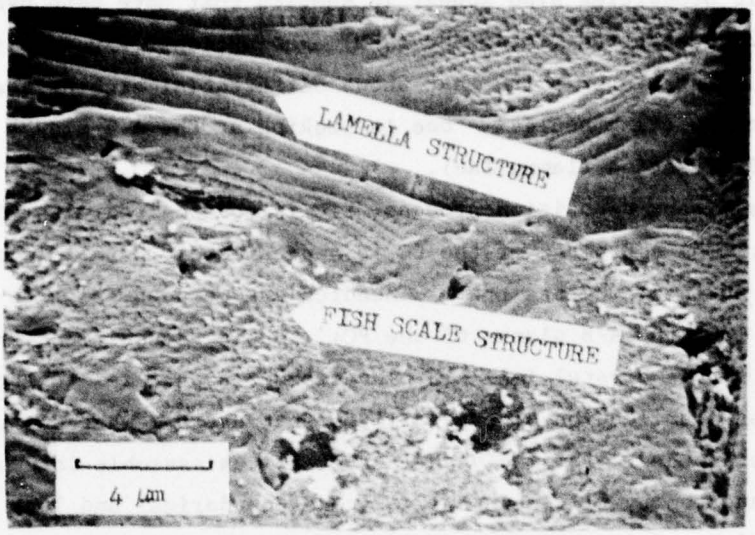


Fig. 6. SEM photomicrograph of substrate graphite (5000X). Hydrogen ion etched for 30 min.

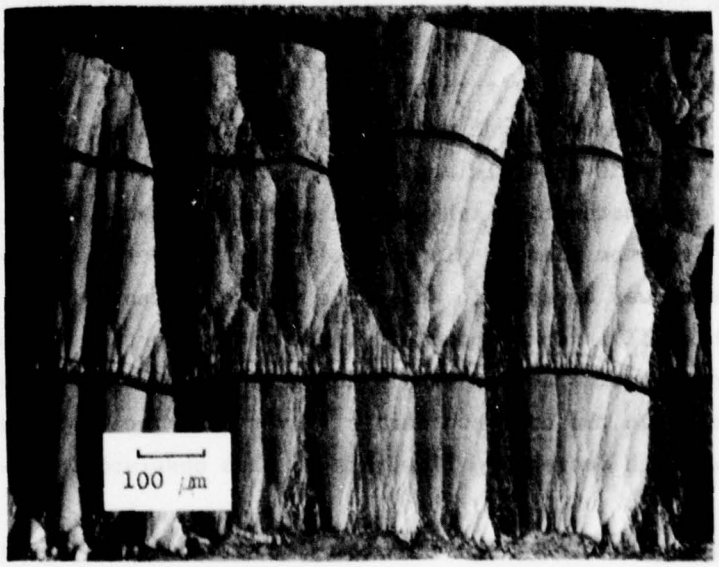


Fig. 7. Photomicrograph (polarized light) illustrating wrinkled appearance and cone-boundaries characteristic of PG (100X).

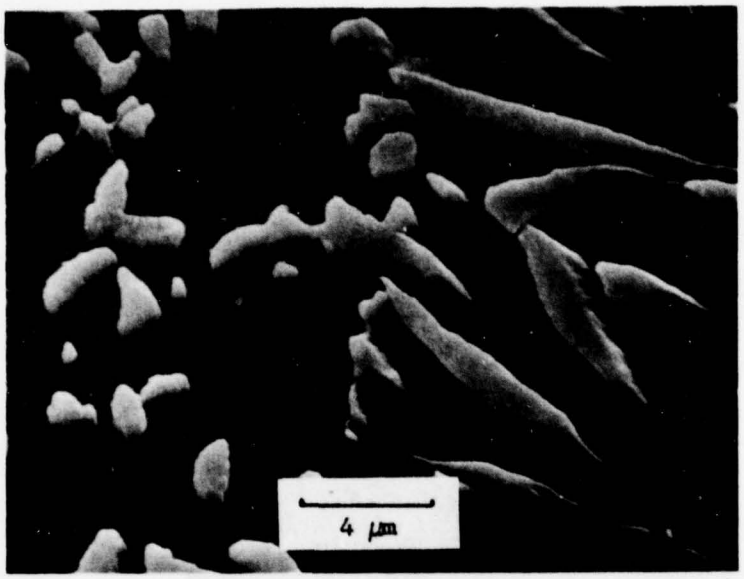


Fig. 8. SEM photomicrograph of adjacent grains at a cone-boundary (5000X). Hydrogen ion etched.

SiC rosette of SiC that caused a lower layer density for a given weight per cent of SiC than coating deposited at higher flow rates.

The microstructure of the codeposited material is very dependent on the substrate on which deposition is initiated. To easily separate layers, thick release coats of PG were deposited between them. The representative microstructure of each layer is not reached until about 1/3 of the layer has been grown. Further, development of a poor structure (rosettes) appears to perpetuate itself into the next layer even though a higher flow rate was used.

In actual fabrication runs, SiC is deposited by itself for a short time before the codeposition of carbon and SiC. This gives a very adherent interface between the substrate and the codeposited layer and a more uniform deispersion of SiC throughout.

IV. DEVELOPMENT OF DEPOSITION PROCESS MODEL

The coating furnace was modeled using the finite-element heat transfer program AYER.[5] This program solves the general two-dimensional equation of heat conduction including effects of time (transient problem), in-plane anisotropic thermal conductivity, and interface thermal contact resistance. Besides the furnace dimensions and material properties, the channel flow furnace model used

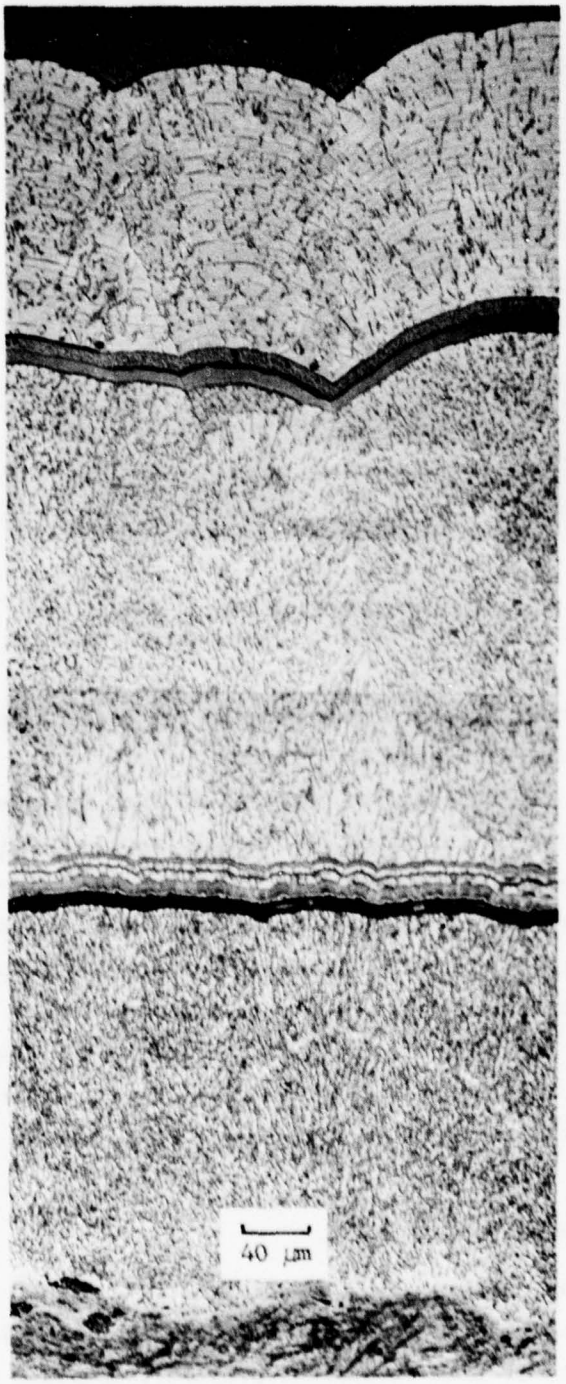


Fig. 9. Photomicrograph of deposited layers (Run 1) 386 mm from plenum (250X). Hydrogen ion etched for 30 min.

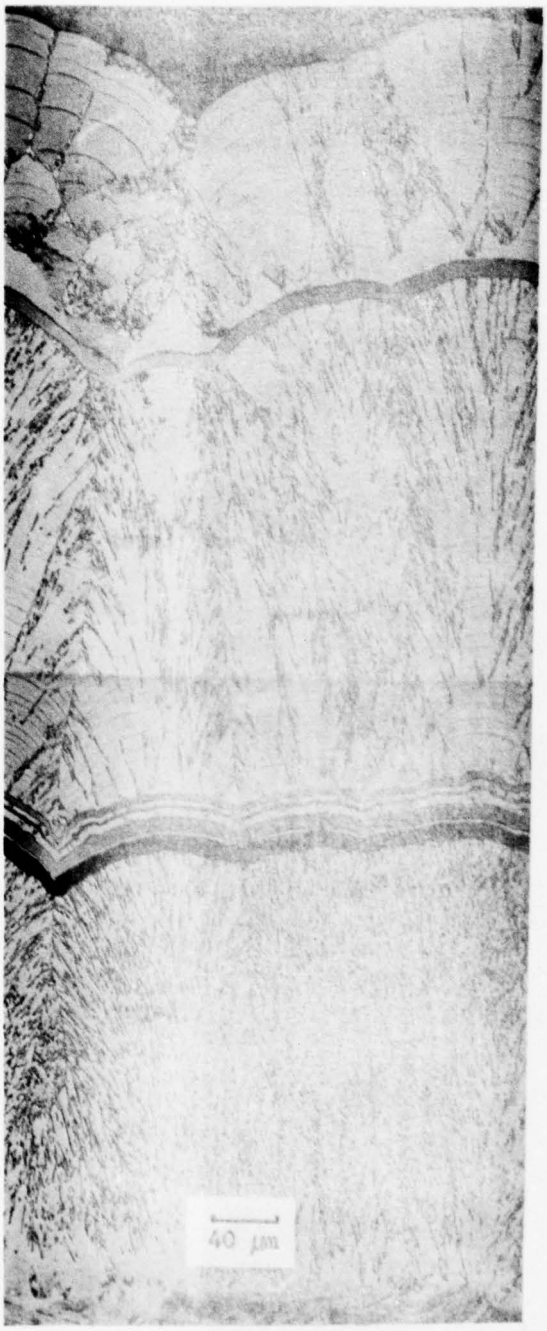


Fig. 10. Photomicrograph of deposited layers (Run 1) 408 mm from the plenum (250X). Hydrogen ion etched for 30 min.

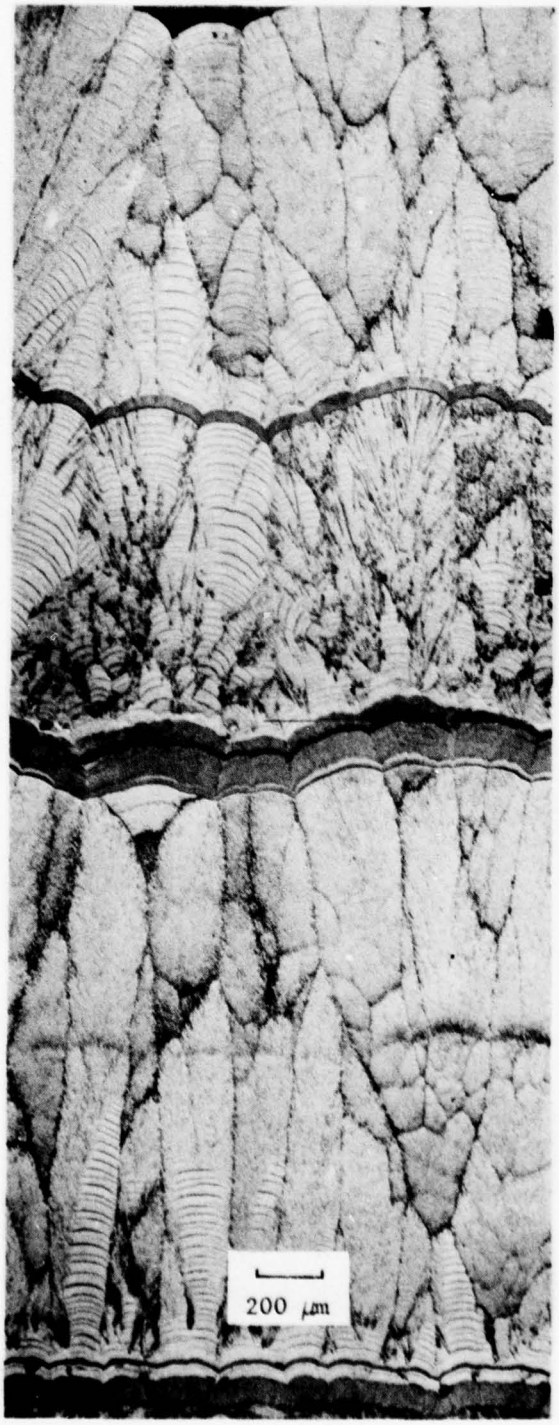


Fig. 11. Photomicrograph of deposited layers (Run 2) 447 mm from the plenum (50X). Hydrogen ion etched.

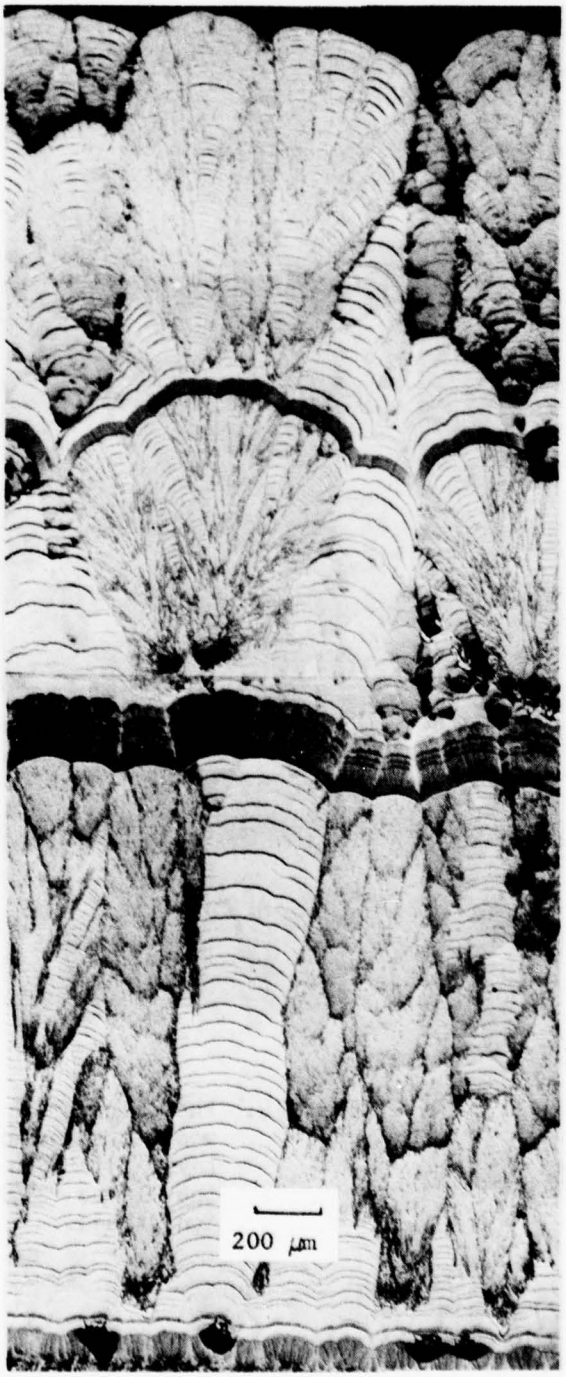


Fig. 12. Photomicrograph of deposited layers (Run 2) 475 mm from the plenum (50X). Hydrogen ion etched.

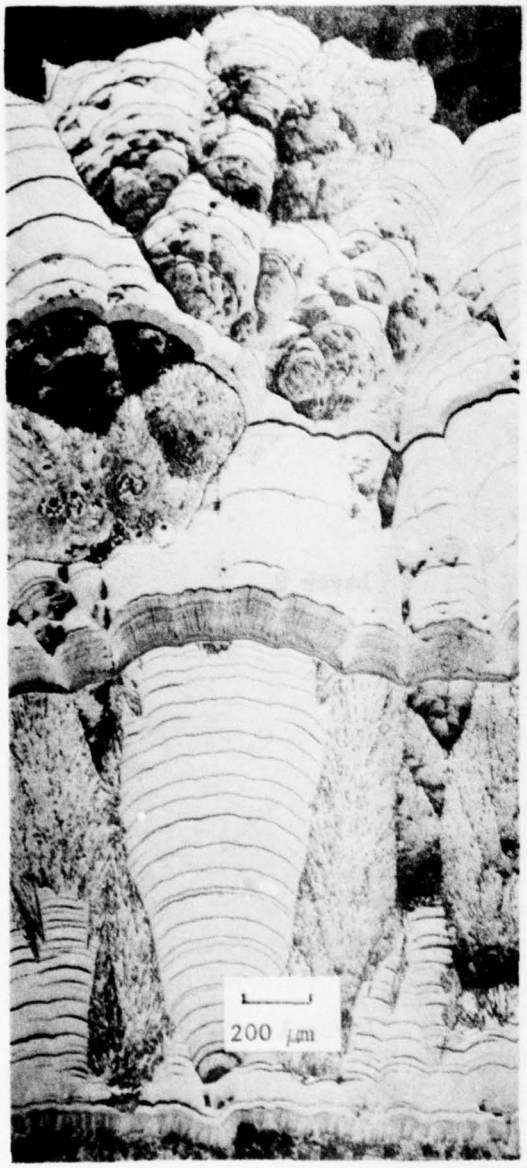


Fig. 13. Photomicrograph of deposited layers (Run 2) 504 mm from the plenum (50X). Hydrogen ion etched.

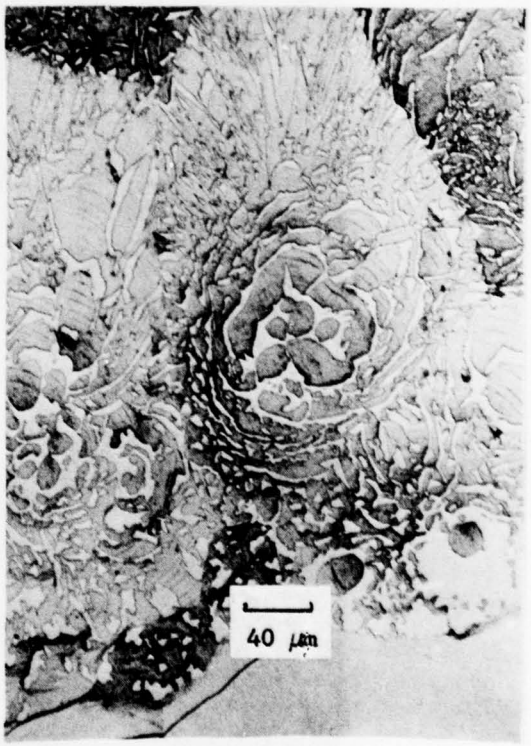


Fig. 14. Photomicrograph of SiC rosette structure (Run 2, Layer 2) at 504 mm from the plenum (250X). Hydrogen ion etched.

the following measured parameters as input.

- Heat generation in the susceptor.
- Water flow rates and temperatures.
- Gas inlet temperature.

AYER output consists of temperatures throughout the model which can be compared with corresponding measurements. Also, the total heat lost to various cooling water circuits is computed and can be used to check an overall heat balance. The calculated temperatures along the flow annulus walls are an input to the fluid dynamics code GENMIX.[2,6] This code solves the two-dimensional, steady parabolic (with second-order velocity differential in one direction only) Navier-stokes equations by marching integration. GENMIX requires as input the measured gas flow rate, temperature, pressure, and velocity distribution at the annulus entrance. When it is supplied with the inlet gas composition, chemical reaction rate constants, and binary diffusion coefficients it can calculate the species concentrations and fluxes in the gas flow. The calculated gas temperatures and axial variation of the wall-to-gas heat transfer coefficient are used as an input to the furnace heat conduction model in AYER. The first part of this section discusses AYER; the second and third parts, GENMIX.

A. AYER Heat Conduction Model

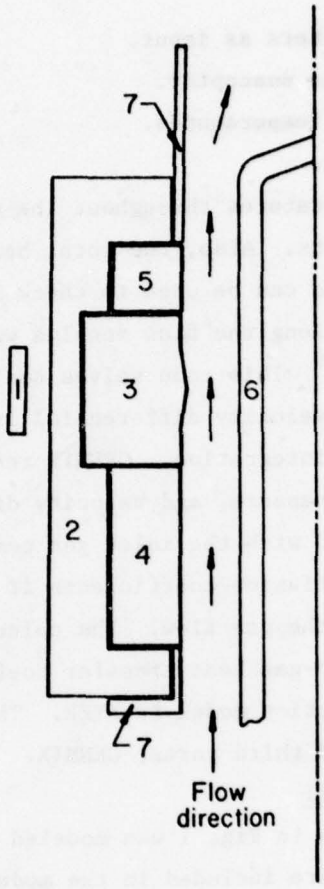
The coating furnace shown in Fig. 1 was modeled as a finite-element mesh, Fig. 15. Only two materials are included in the model, ATJ graphite (parts 3, 4, 5, and 7) and carbon or graphite felt insulation (part 2). The water-cooled copper center body and induction coil are included to illustrate their relative positions.

Between the first and second coating runs the furnace and the AYER model were modified as follows.

- A 13 mm-thick horizontal layer of graphite felt was installed between the substrate ring and the spacer rings (Fig. 15). The felt extended from the o.d. to within 9.5 mm of the i.d. of the graphite spacer rings.
- The mica wrapping and one layer of felt were removed from under the induction coil.

These changes were made because the first coating run showed that the methane gas was being heated too rapidly and the coil was beginning to arc through the mica to the felt.

1. Power Generation. The power generated in the model is distributed in



1 Induction coil
2 Carbon felt insulation
3 Substrate ring, ATJ graphite
4 Bottom spacer ring, ATJ graphite
5 Top spacer ring, ATJ graphite
6 Copper center body
7 Upper and lower flow directors, ATJ graphite

Fig. 15. AYER model of coating furnace.

the outer 19 mm of the ATJ substrate ring; under the coil, the outer 13 mm generates heat at twice the rate per unit volume that the inner 6 mm does. The power drops to zero very quickly at the ends of the coil. The total power is obtained from the measured power input to the furnace minus the losses in the cooling water (see Table III).

2. Material Properties. ATJ graphite properties were obtained from Ref. 7. The carbon and graphite felt, called "UCAR" is a product of Union Carbide Corporation. Supplier's literature dated 1974 contained density, emissivity, and specific heat data for this material. The thermal conductivity was presented as an approximate effective value when the cold surface is held at approximately 311 K (100°F). The average temperature of the material is then some weighted value between 311 K and the hot-face temperature that was used as a parameter in the supplier's literature. These approximate data were converted to thermal conductivity values at the average felt temperature to conform to the requirements of the computer program. After comparing measured and calculated temperatures for the nitrogen flow tests, the approximate thermal conductivity was increased by a factor of 2 and agreement with measured values was good. After 24 h, the carbon felt was visibly shrunken and compacted. An additional 25% increase in thermal conductivity improved the agreement near the end of a coating run, apparently because of the shrinkage.

3. Boundary Conditions.

a. Gas flow correction. The axial distribution of gas temperature and heat-transfer coefficient was obtained from the flow model, GENMIX.

The heat pickup by the gas was about 1.5 kW, both by analysis and measurement. This is only 3-4% of the susceptor power, in contrast to approximately 36% in the injector deposition.

b. Radiation to the center body. The water-cooled copper center body is one of the largest furnace heat sinks, accounting for about 25 kW or 45% of the susceptor power. This heat loss is highly dependent on the thermal emissivity of the copper. The emissivity used for the dull-finish copper at 450 K was 0.42. Lower emissivities, down to 0.05 could be justified for the mechanically cleaned and sandpapered copper as it was put into the furnace. From the measured temperatures, it is believed that the copper becomes discolored or oxidized during the heatup phase of furnace operation. The radiation is calculated as a grey body, with a geometric view factor of unity.

c. Radiation and natural convection to bell jar, etc. Thermal radiation and free convection will take place between the hot and cool surfaces inside the bell jar. An exact treatment is difficult because of the many different surface conditions, orientations, and temperatures. The natural convection was calculated by a standard correlation* and the thermal radiation was determined from published emissivity values. The thermocouple leads and coil supports were neglected in calculating radiation view factors between the felt and the bell jar. Although it could be modified to do so, the model does not differentiate between the bell jar, bell jar base, and coil support. The total water heat pickup in these three components is about 13 kW or 23% of the susceptor power. It was expected that an empirical adjustment of the bell jar boundary condition would be required because of the simplifications mentioned. After the modification in the felt thermal conductivity to match the temperature at T-5, no further adjustment was required.

d. Radiation and conduction to coil-18000 Series Tests. In addition to the heat pickup in the coil cooling water from electrical losses, about 10-14 kW of power is transferred from the felt outer diameter by radiation and conduction through stagnant nitrogen.** The gap between the felt and coil is small enough that natural convection was not significant. The felt emissivity is 0.99 as recommended by the supplier. The coil i.d. stayed relatively clean during the tests, and an emissivity value of 0.21 was used. The method is described in Vol. I, Section IV.A.3.d. The calculated heat transfer by thermal radiation was about three times the amount transferred by conduction in stagnant nitrogen.

In the 1600 and 1700 series tests, the outer surface of the felt was in contact with the coil, except for a thin (0.5 mm) mica electrical insulator. The boundary condition was the coil cooling water temperature and the convective heat transfer coefficient, obtained from standard formulas. Surprisingly, the calculated heat lost to the coil was about the same (10-14 kW) as in the 18000 series test. Pretest predictions by the AYER code had indicated that the heat pickup would be about 2.5 kW higher in the 18000 series for the same substrate temperature. The apparent discrepancy is probably caused by the fact that the 18000 series ran with a 100 K cooler substrate temperature than the earlier tests and the pretest prediction.

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* See Vol. I, Section IV. A.3.

** The felt was in contact with coil for the 16000 and 17000 tests.

e. Loss to lower precooler. The water-cooled lower precooler is heated by conduction from the graphite part adjacent to the lower flow deflector. The boundary condition was simplified by summing the two series resistances; conduction through the brass and convection to the cooling water. The convection coefficient was obtained from a standard formula by using the measured water flow and the dimensions of the flow channels. The measured and calculated losses to the precooler were 3.1 kW or 5% of the susceptor power.

4. Effect of Coating Layers. The AYER model was modified to account for the effect of coating layers 1 and 2 for both coating tests. This was accomplished by increasing the thickness of the finite elements by an amount equivalent to the layer's measured thickness and adjusting the thermal conductivity values in those elements to account for the reduced conductivity of the codeposit. The PG/SiC conductivity was obtained from data published[8] by ARC. The conductivity depends on the SiC content and (probably) on the microstructure. An average value for the SiC content of layers 1 and 2 in each coating test was used to obtain the conductivity. There are no data of microstructure effects. The results of the model predictions for the coating runs are presented and compared with measurements in Sec. III B.

B. Gas Flow, Heat Transfer, and Chemical Kinetics Model.

The geometry of the LASL coating furnace described here produces a channel-type flow. The flow remains attached to the walls and has no regions of recirculation. The experimental flow rates in the furnace produced a range of Reynolds numbers that indicate both laminar and turbulent flows. Therefore, provision was made for modeling both laminar and turbulent flows with heat transfer and chemical reactions.

1. GENMIX Code. The GENMIX code contains a numerical solution method that can solve heat and mass transfer in boundary-layer-type flow. The code can solve momentum, heat, and mass transfer in steady, two-dimensional, boundary layer flow. The flow may be laminar or turbulent and may include chemical reactions. In the boundary layer approximation, the gradients are assumed to be othogonal to the flow stream lines. Therefore, the solution procedure is not applicable to a recirculating flow, because the flow is then described by elliptic rather than parabolic partial differential equations. The solution is obtained for points at a particular longitudinal station based on previously calculated upstream values, before stepping downstream to repeat the procedure.

The code operation was checked by computing solutions to heat transfer and

chemical kinetic problems for which previously obtained solutions were known.[9,10] The first check case was a constant heat-rate entry length problem for a gas with constant properties, and no chemical reactions. The fluid was assumed to enter the annulus with a uniform velocity and temperature profile. The inner wall was assumed insulated, a constant heat flux was applied at the outer wall, and laminar flow was assumed.

A second nonreacting check case was also solved using the code. In this case, the inner wall was also insulated and a constant heat flux was applied at the external wall, but the flow was turbulent with a Reynolds number of 5000. The velocity profile was assumed to be fully developed and the temperature profile was constant at the entrance.

The results of these two check cases are shown in Fig. 16 with the corresponding comparison solutions. The Nusselt number based on longitudinal distance from the inlet is plotted versus the nondimensionalized distance from the inlet. The results compare very favorably through the entry region and into the region of fully developed velocity and temperature.

The calculation of the chemical source terms in the code was checked by programming a problem in which a uniform concentration of CH_4 entered the annulus and reacted to C_2H_6 . The good agreement between computed and analytical results and the results of the previously described check cases gave confidence that the results calculated by the code for the furnace configuration would be correct.

2. Flow and Heat Transfer Model. The gas mixture entered the furnace from a plenum below the heated section. The gas was well mixed in the plenum and was assumed to enter the annular flow passage with uniform velocity, temperature, and species concentrations.

The velocity boundary conditions applied at the walls were zero velocity in the axial and transverse directions. The inner and outer wall temperatures were input from calculations obtained using the AYER heat transfer code, described in Sec. IV. A.

3. Chemical Kinetics Model. The postulated chemical kinetics model for methane pyrolysis is

$$CH_4 \xrightarrow{K_1} \tfrac{1}{2}C_2H_6 \xrightarrow{K_2} C_2H_4 \xrightarrow{K_3} C_2H_2 \xrightarrow{K_4} \tfrac{1}{2}C_4H_2 \rightarrow \text{deposition} \qquad (6)$$

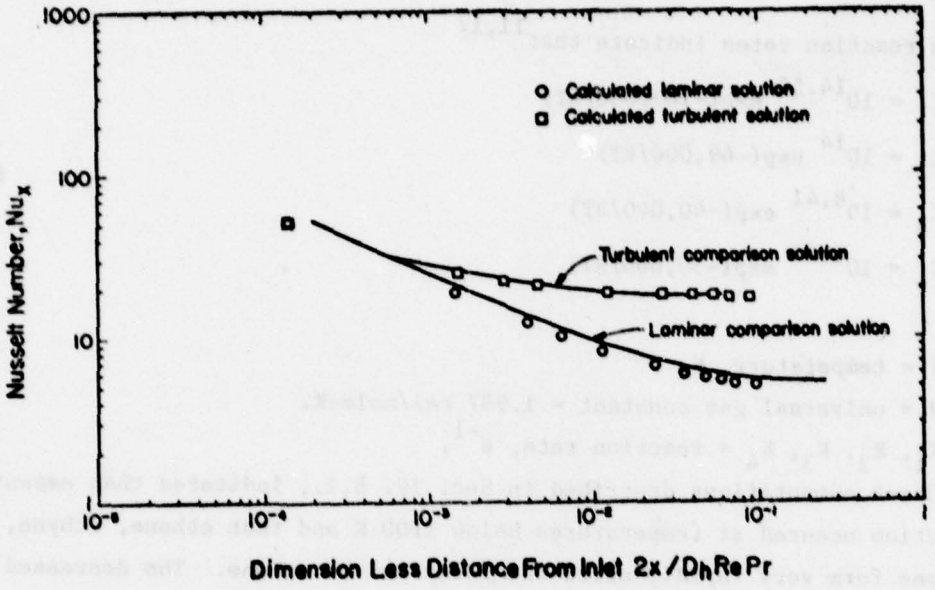

Fig. 16. Entry region solutions obtained for laminar and turbulent nonreacting flows.

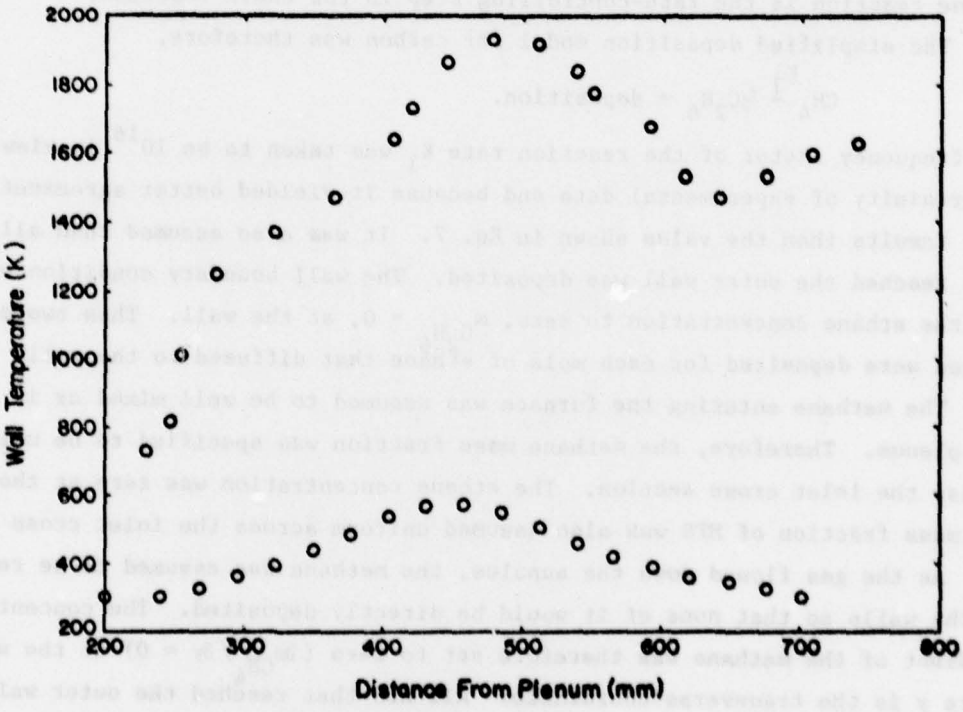

Fig. 17. Inner and outer wall temperatures.

Data on reaction rates indicate that[11,12]

$$K_1 = 10^{14.58} \exp(-103{,}000/RT)$$
$$K_2 = 10^{14} \exp(-69{,}000/RT)$$
$$K_3 = 10^{8.41} \exp(-40{,}000/RT) \quad (7)$$
$$K_4 = 10^{6.23} \exp(-30{,}000/RT)$$

where

T = temperature, K.

R = universal gas constant = 1.987 cal/mole-K.

K_1, K_2, K_3, K_4 = reaction rate, s^{-1}.

Check computations described in Sec. IV. B.1., indicated that essentially no reaction occured at temperatures below 1500 K and that ethene, ethyne, and butadiyne form very rapidly after the formation of ethane. The decreased activation energies for succeeding steps in the chain reaction more than offset the decreasing frequency factors, so that the reaction rates K_2, K_3, K_4 are much higher than the rate of reaction K_1 of methane to ethane. Thus, the methane-ethane reaction is the rate-controlling step in the chain reaction.

The simplified deposition model for carbon was therefore,

$$CH_4 \xrightarrow{K_1} \tfrac{1}{2}C_2H_6 \rightarrow \text{deposition.} \quad (8)$$

The frequency factor of the reaction rate K_1 was taken to be 10^{16} in view of the uncertainity of experimental data and because it yielded better agreement with test results than the value shown in Eq. 7. It was also assumed than all ethane that reached the outer wall was deposited. The wall boundary condition was to set the ethane concentration to zero, $m_{C_2H_6} = 0$, at the wall. Thus two moles of carbon were deposited for each mole of ethane that diffused to the wall.

The methane entering the furnace was assumed to be well mixed as it left the plenum. Therefore, the methane mass fraction was specified to be uniform across the inlet cross section. The ethane concentration was zero at the inlet. The mass fraction of MTS was also assumed uniform across the inlet cross section.

As the gas flowed down the annulus, the methane was assumed to be reflected at the walls so that none of it would be directly deposited. The concentration gradient of the methane was therefore set to zero ($\partial m_{CH_4} / \partial y = 0$) at the wall, where y is the transverse coordinate. All MTS that reached the outer wall was assumed to be deposited as SiC, similar to the coundary condition for C_2H_6.

4. Input Data. Input data supplied to GENMIX included flow rates and properties of the gases, wall temperatures calculated with the AYER heat transfer code, and nodal point spacing to achieve desired numerical accuracy with efficient use of computer time.

According to the kinetic-molecular theory of gases, the viscosity of an ideal gas is proportional to the square root of the temperature.[13] The relation was fit to experimental data for N_2.[14] The nitrogen mass fraction of the gas mixture exceeded 0.93 in all cases; therefore, the properties approximated those of nitrogen. For laminar flow, the viscosity was determined from

$$\mu = 1.307 \times 10^{-6} \sqrt{T} \tag{9}$$

where T = temperature (K) and μ = absolute viscosity (Ns/m^2).

When the flow is turbulent, the gas transport properties are enhanced by turbulent mixing of the fluid. The effect of turbulence was modeled by calculating an effective viscosity, caused by turbulence based on an approximation to the van Driest formula for modeling turbulence near walls, which was added to the laminar viscosity.

The nitrogen thermal conductivity was input by specifying the Prandtl number. The Prandtl number was determined by evaluating the nitrogen properties at 1800 K, because chemical reactions and deposition occur only above 1500 K and the maximum wall temperature in the furnace was 2030 K. Thus, the Prandtl number was set equal to 0.8147.

Diffusion coefficients, D, for CH_4, C_2H_6, and MTS in nitrogen were not input directly, but were supplied by specifying the Schmidt numbers, Sc, for each constituent gas. From the definition of the Schmidt number, Sc,

$$D = \mu/\rho \, Sc. \tag{10}$$

For a perfect gas and constant Sc, Eq. 9 can be substituted into Eq. 10 to obtain

$$D = 1.307 \times 10^{-6} \, RT^{3/2}/W \, p \, Sc. \tag{11}$$

where R is the universal gas constant, p is the pressure, and W is the molecular weight. Under these conditions, Eq. 11 shows that $D \sim T^{3/2}$. The correlation agrees well with experimental data.[15,16] The viscosity, density, and diffusion coefficients were evaluated at 1800 K to give a good data fit in the outer wall region. These values were used to calculate the input Sc for each chemical species.

Thus,

$$Sc_{CH_4} = 0.3714,$$
$$Sc_{C_2H_6} = 0.9506, \quad (12)$$
$$Sc_{MTS} = 1.269.$$

In turbulent flow, the laminar Pr and Sc numbers are small compared to the effective Pr and Sc numbers because of turbulent eddies. Experimental measurements have shown[17] that near a wall the effective Pr is very nearly 1. Therefore, in modeling those deposition runs where the flow was turbulent, the Pr and Sc numbers were set equal to unity.

The wall temperatures were calculated by iterating twice using the AYER heat transfer code and GENMIX. The temperatures supplied as boundary coditions for the second coating run are shown in Fig. 17. Data were input at the points shown, and linear interpolation was used to evaluate wall temperatures at distances between data points. The inner wall temperature is so much lower than that at the outer wall that it has a negligible effect on deposition.

Because chemical reactions occur only near the outer wall, the outer wall nodal points were more closely spaced, thus producing higher accuracy.

5. Results. The results of the flow, heat transfer, and chemical kinetics model are shown in Figs. 18-29. Figures 18-22 apply to conditions when Layer 1 of Run 2 was being deposited. The gas stream velocity as a function of transverse distance from the external wall is shown in Fig. 18. The entrance Reynolds number is 1186, well below the critical Reynolds number for turbulent flow. The velocity is shown for four axial distances from the plenum where the flow is viewed as being from left to right. The development of the velocity profile as it flows through the furnace can be seen. The flow is asymmetric about the center of the gap, because of variable fluid properties. At the exit from the furnace, the flow is still not fully developed.

Figure 19 shows the development of the temperature profile as the gas flows through the furnace. The major temperature variation is confined to the outer half of the flow stream. The temperature variation at the inner wall is relatively minor. Even as the gas leaves the furnace, inner and outer thermal boundary layers just barely meet to cover the full width of the flow channel. At 600 mm from the plenum, both the inner and outer wall temperatures have decreased from their maxima, thus causing the cross-over of the temperature profiles

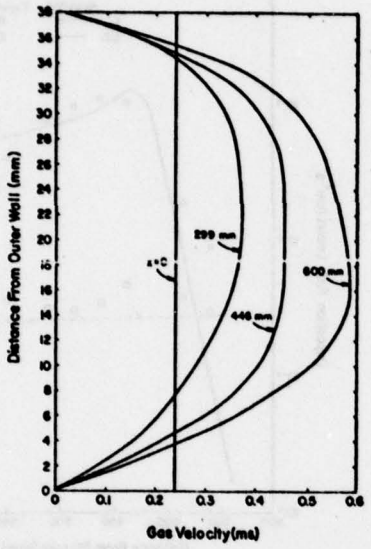

Fig. 18. Velocity as a function of transverse distance. Run 2, Layer 1.

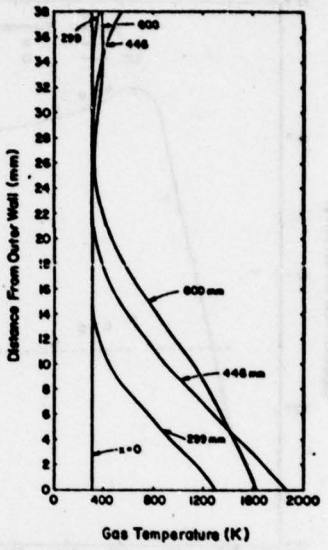

Fig. 19. Temperature as a function of transverse distance. Run 2, Layer 1.

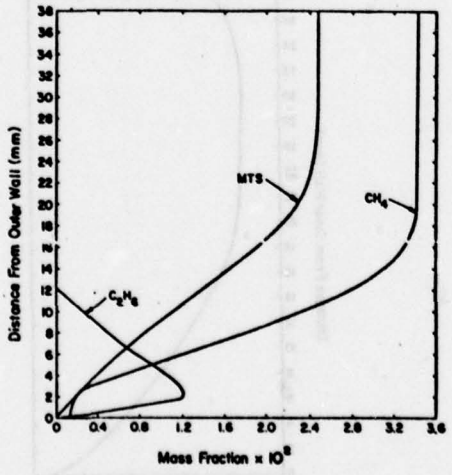

Fig. 20. Species mass fraction as a function of transverse distance at 446 mm from plenum. Run 2, Layer 1.

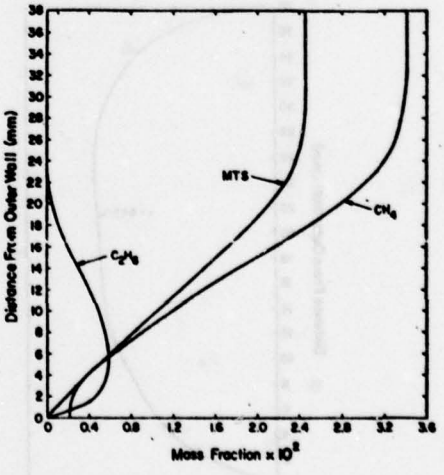

Fig. 21. Species mass fraction as a function of transverse distance at 600 mm from plenum. Run 2, Layer 1.

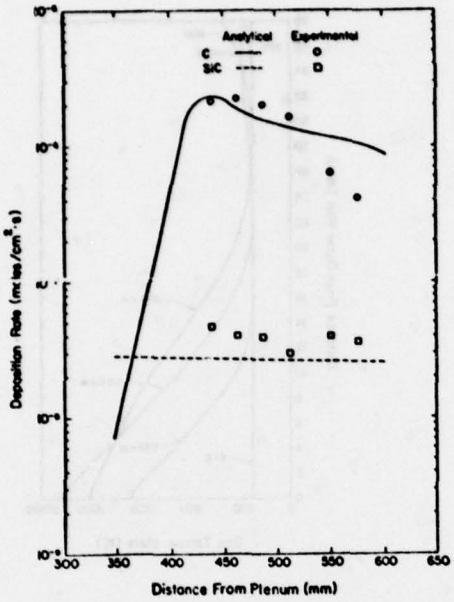

Fig. 22. Calculated and experimental deposition rates as a function of longitudinal distance. Run 2, Layer 1.

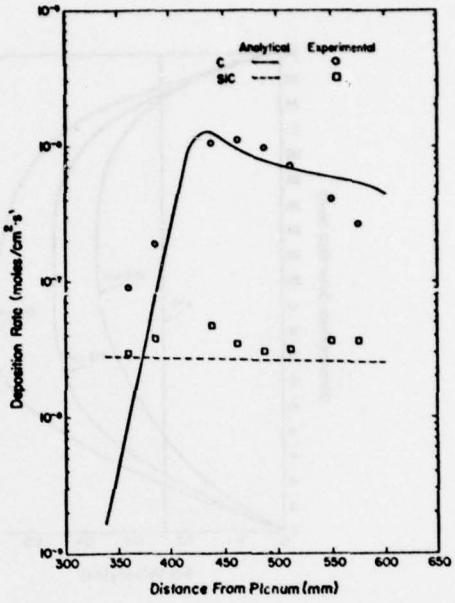

Fig. 23. Calculated and experimental deposition rates as a function of longitudinal distance. Run 2, Layer 2.

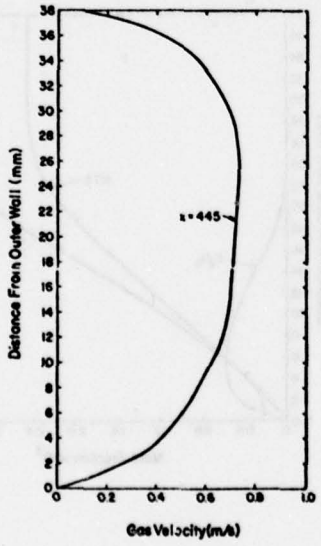

Fig. 24. Velocity as a function of transverse distance at 445 mm from plenum. Run 2, Layer 3.

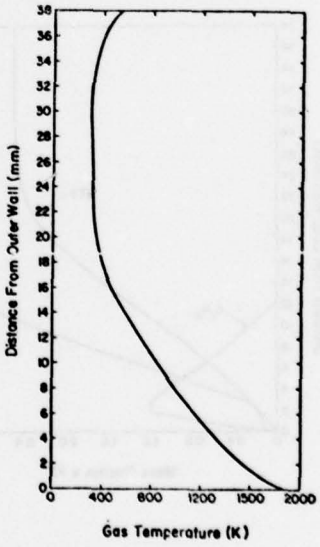

Fig. 25. Temperature as a function of transverse distance at 445 mm from plenum. Run 2, Layer 3.

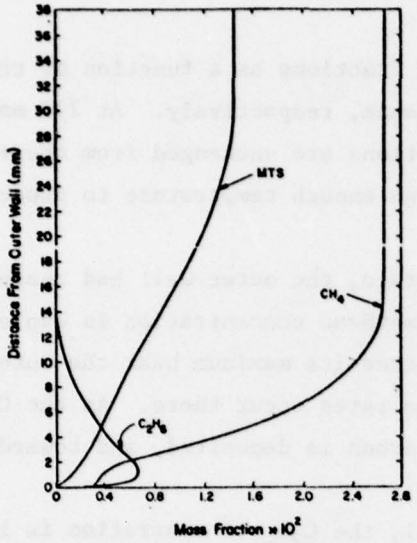

Fig. 26. Species mass fraction as a function of transverse distance at 445 mm from plenum. Run 2, Layer 3.

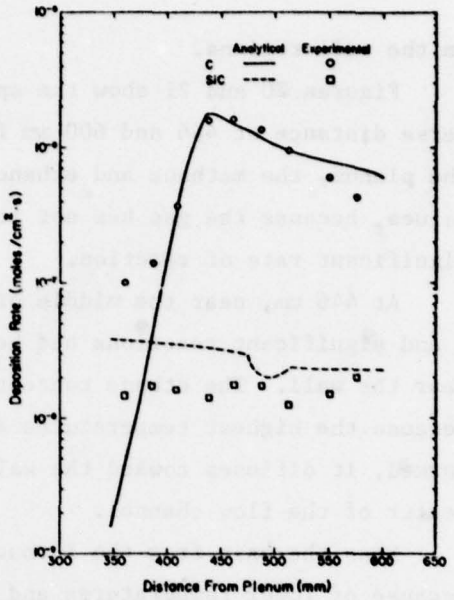

Fig. 27. Calculated and experimental deposition rates as a function of longitudinal distance. Run 2, Layer 3.

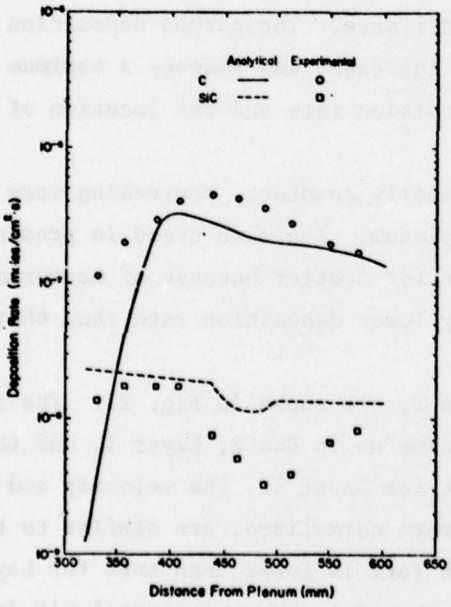

Fig. 28. Calculated and experimental deposition rates as a function of longitudinal distance. Run 1, Layer 1.

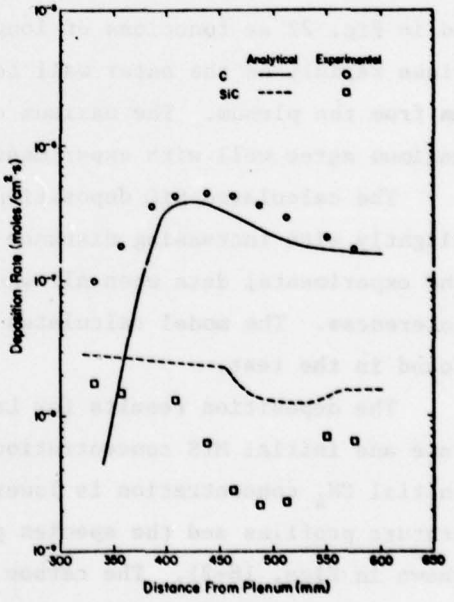

Fig. 29. Calculated and experimental deposition rates as a function of longitudinal distance. Run 1, Layer 2.

in the wall regions.

Figures 20 and 21 show the species mass fractions as a function of transverse distance at 446 and 600 mm from the plenum, respectively. At 299 mm from the plenum, the methane and ethane concentrations are unchanged from their initial values, because the gas has not reached a high enough temperature to produce a significant rate of reaction.

At 446 mm, near the middle of the substrate, the outer wall had reached 1630 K and significant reactions had begun. The methane concentration is depleted near the wall. The ethane concentration reaches its maximum near the outer wall, because the highest temperatures and reaction rates occur there. As the C_2H_6 is formed, it diffuses toward the wall, where carbon is deposited, and toward the center of the flow channel.

Near the exit from the furnace (Fig. 21), the C_2H_6 concentration is lower because of lower temperatures and resulting lower rates of formation. The regions of significant C_2H_6 concentration and depleted CH_4 and MTS concentration extend past the center of the flow channel because of increased diffusion time and higher temperatures within the flow stream.

The calculated and experimental deposition rates of carbon and SiC are plotted in Fig. 22 as functions of longitudinal distance. The carbon deposition rate rises rapidly as the outer wall temperature increases and reaches a maximum 450 mm from the plenum. The maximum carbon deposition rate and the location of the maximum agree well with experiment.

The calculated SiC deposition rate is nearly constant, decreasing very slightly with increasing distance from the plenum. The same trend is present in the experimental data when allowance is made for scatter because of measurement tolerances. The model calculates a slightly lower deposition rate than that found in the test.

The deposition results for Layer 2, Run 2, are shown in Fig. 23. The flow rate and initial MTS concentration are the same as in Run 2, Layer 1, and the initial CH_4 concentration is lower than that for Layer 1. The velocity and temperature profiles and the species profiles, when normalized, are similar to those shown in Figs. 18-21. The carbon deposition rate is lower than that for Layer 1 because of lower initial concentrations. Both model and experimental SiC deposition rates agree very well with Layer 1 results.

Layer 3 of Run 2 was deposited at a higher flow rate, which resulted in a

Reynolds number slightly greater than 2000 and placed the flow in the transition region from laminar to turbulent. Comparison of model calculations with test results showed that the flow was turbulent. The Reynolds number is defined as $Re = \rho U D_h/\mu$. The product $\rho U D_h$ is nearly constant because the cross-sectional geometry is constant through the furnace. There is a loss of mass from deposition at the outer wall, but it is small compared to the overall flow rate. The viscosity, however, varies as $\sqrt{T}$ and increases markedly as the gas flows through the furnace, particularly in the outer wall region. Therefore, Re decreases with distance from the plenum, and relaminarization or reverse transition from turbulent to laminar flow may occur at some point in the furnace. Figures 24-26 show velocity, temperature, and species mass fraction as functions of transverse distance from the outer wall at 445 mm from the plenum, which is near the center of the substrate. The deposition rates of carbon and SiC as functions of axial distance are shown in Fig. 27. The carbon deposition rate agrees very well with the experimental data. The calculated rate of SiC deposition is higher than the rates for Layer 2, even though the initial concentration of MTS is lower. The higher deposition rate is caused by higher effective diffusion coefficients from turbulent mixing. The combination of low MTS concentration and increased diffusion rates appears to deplete the MTS concentration in the wall region, thus causing lower deposition rates in the region from 475 to 525 mm from the plenum.

All three layers of Run 1 were deposited at flow rates in the turbulent regime, so Run 1 is dicussed last. The initial concentrations of CH_4 and MTS were lower than for Run 2. The velocity, temperature, and species profiles, when normalized, are similar to those for Layer 3, Run 2, shown in Figs. 24-26. The deposition rates as function of longitudinal distance are shown in Figs. 28 and 29 for Layers 1 and 2, respectively. The lower initial concentration of MTS causes a decrease in SiC deposition rate in both cases. This relative change is evident in both the model and test results and supports the hypothesis that the MTS concentration becomes depleted in the wall region.

The linear dependence of maximum carbon deposition rate on the initial concentration of CH_4, which was discovered in the experimental work, is predicted well by the model. The rates calculated by the model and the furnace test results are compared in Fig. 30.

6. Conclusions. A satisfactory mathematical of the flow, heat transfer, and chemical kinetics of the carbon and SiC deposition in the coating furnace

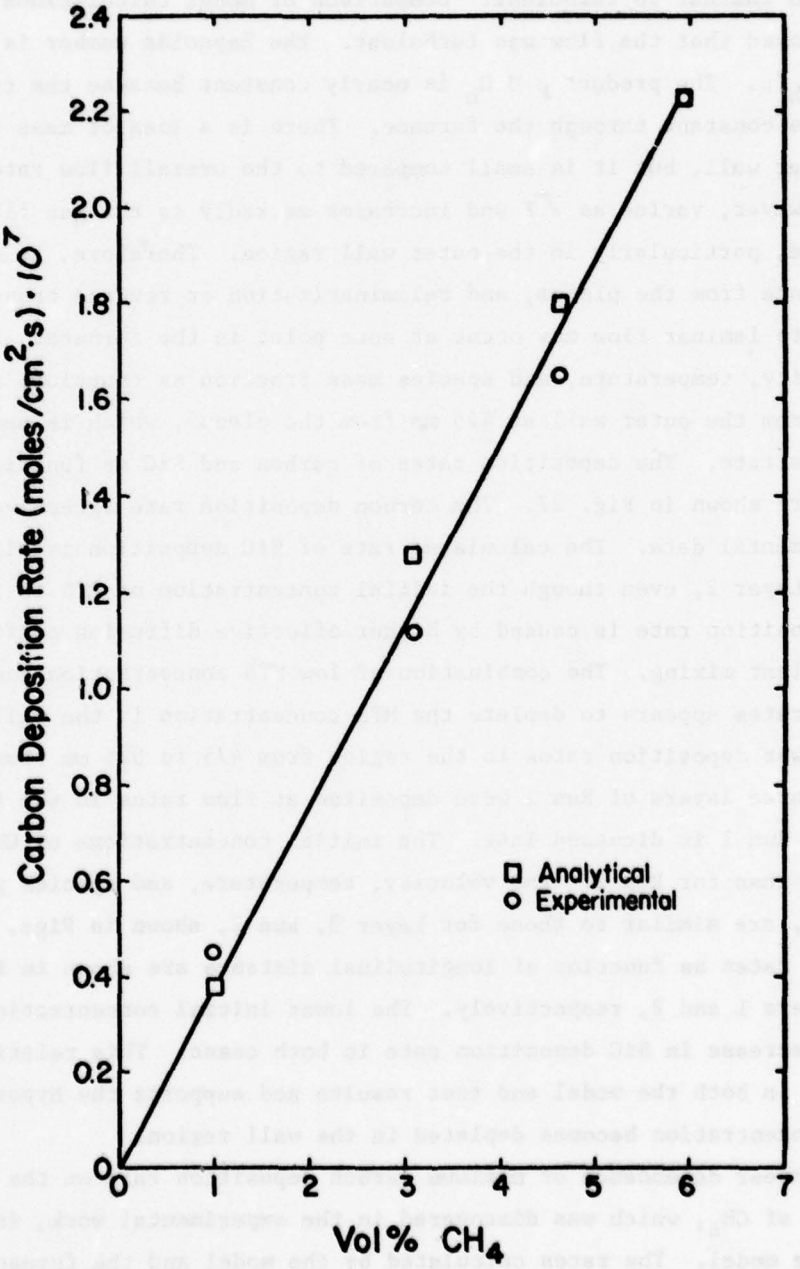

Fig. 30. Maximum carbon deposition rate dependence on initial methane concentration.

has been developed. Model and test results agree very well. The results were computed in very little computer time, the final run of each case requiring approximately 45 s on a CDC 6600.

Parametric studies of furnace and flow characteristics can be carried out using the model in its present form. Thus, the code can be used to model the deposition characteristics of other furnaces before they are built, resulting in a great savings of time and funds in future PG/SiC deposition programs.

V. SUMMARY

A channel flow CVD furnace was designed, built, instrumented, tested, and analyzed. The PG/SiC coating's microstructure and inferred quality were characterized by metallographic methods and were correlated with the process variables.

The maximum PG deposition rate increases as a linear function of the initial concentration of CH_4, as verified both experimentally and analytically. The PG deposition rate is controlled by the decomposition of CH_4 to C_2H_6, which occurs at temperatures above 1500 K. The axial distribution of PG on the substrate can be modified by changes in the wall temperature in the coating chamber.

The experimental maximum SiC deposition rate appears to increase linearly with increasing MTS concentration in the process gas, but the data were more scattered than those for PG and the analytical model did not confirm the slope. For a given MTS concentration, the SiC deposition rate is relatively constant with axial distance from the inlet. When the initial MTS concentration is relatively low and mass transfer rates are high (i.e. with turbulent flow), the MTS becomes depleted in the wall region, causing a dip in the deposition rates between 475 and 525 mm from the plenum.

Metallography of the coating confirmed that the microstructure changes from a uniform distribution of accicular SiC within the grains (the desired quality) to coarse SiC crystals concentrated at the grain boundaries (undesirable) as the flow rate of process gas is decreased. The microstructure is very dependent on the substrate on which nucleation is initiated, and poor structure can perpetuate itself into layers where, ordinarily, a better quality coating would be expected.

An analytical model of the channel-flow coating furnace was developed by use of two general-purpose computer programs, AYER and GENMIX. Model predictions and experimental results agreed very well. Parametric studies of this furnace

and its flow characteristics can be carried out with the model in its present form. The computer codes can be used to model the deposition characteristics of other furnaces before they are built, to save time and funds.

ACKNOWLEDGMENTS

We appreciate the interest and advice of Major J. G. Dean, the Air Force Rocket Propulsion Laboratory Technical Project Officer for this program. We also acknowledge the work of R. D. Reiswig and S. S. Levinson on optical and scanning electron microscopy; and the efforts of C. Hollabaugh, L. A. Dalton, C. W. Mick, J. C. Miller, and C. Radosevich in the performance of the heating and coating tests.

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APPENDIX A

COATING FURNACE AND PROCESS EQUIPMENT

I. GENERAL

The furance assembly was installed in an existing water-jacketed bell jar (nominal 1-m i.d.) modified for the coating application (Fig. A-1). The lower section of the bell jar, the (plenum chamber), contained the gas distribution manifold and the center body mounting support (Fig. A-2). A water-jacketed base plate above the manifold separated the plenum chamber from the upper suctions and supported the furnace precooler and heating coil assemblies (Fig. A-3). A water-cooled center body extended from the bottom of the plenum chamber through an orifice in the base plate into the coating region. The coating gas passed through the space between the center body and the furnace precooler and graphite ring assemblies up to the water-cooled exhaust canopy. From the exhaust canopy, the gas is piped through a heat exchanger (Fig. A-4) and then discharged into a filtered laboratory exhaust air duct.

The bell jar could be operated under vacuum by valving off the process gas supply and the exhaust system and opening the appropriate valves to an oil diffusion pump and mechanical forepump system. This mode of operation permitted vacuum outgassing (at temperature) of the carbon felt insulation before the processing runs.

Power for the induction coil was provided by a 150 kW motor-generator set at 10 kHz through a vacuum-tight coaxial power port in the center bell jar section (Fig. A-1). Vacuum-tight fittings were installed in flanges attached to bell jar ports to provide water and instrumentation connections (Figs. A-1 and A-4). The exhaust gases were analyzed using a quadropole gas analyzer. A sampling tube brought part of the exhaust gas stream into a differential pumped chamber that was connected to the house vacuum system. Part of the differential pumped chamber of the atmosphere was leaked into the higher vacuum gas analysis chamber of the quadropole mass spectrometer.

II. SERVICES

A. Process Nitrogen

Process nitrogen was provided by a high-pressure compressed gas trailer (45,000 SCF). The gas passed through a pressure-reducing regulator, connecting line, mass flow rate transducer, and control valve, and then into the gas

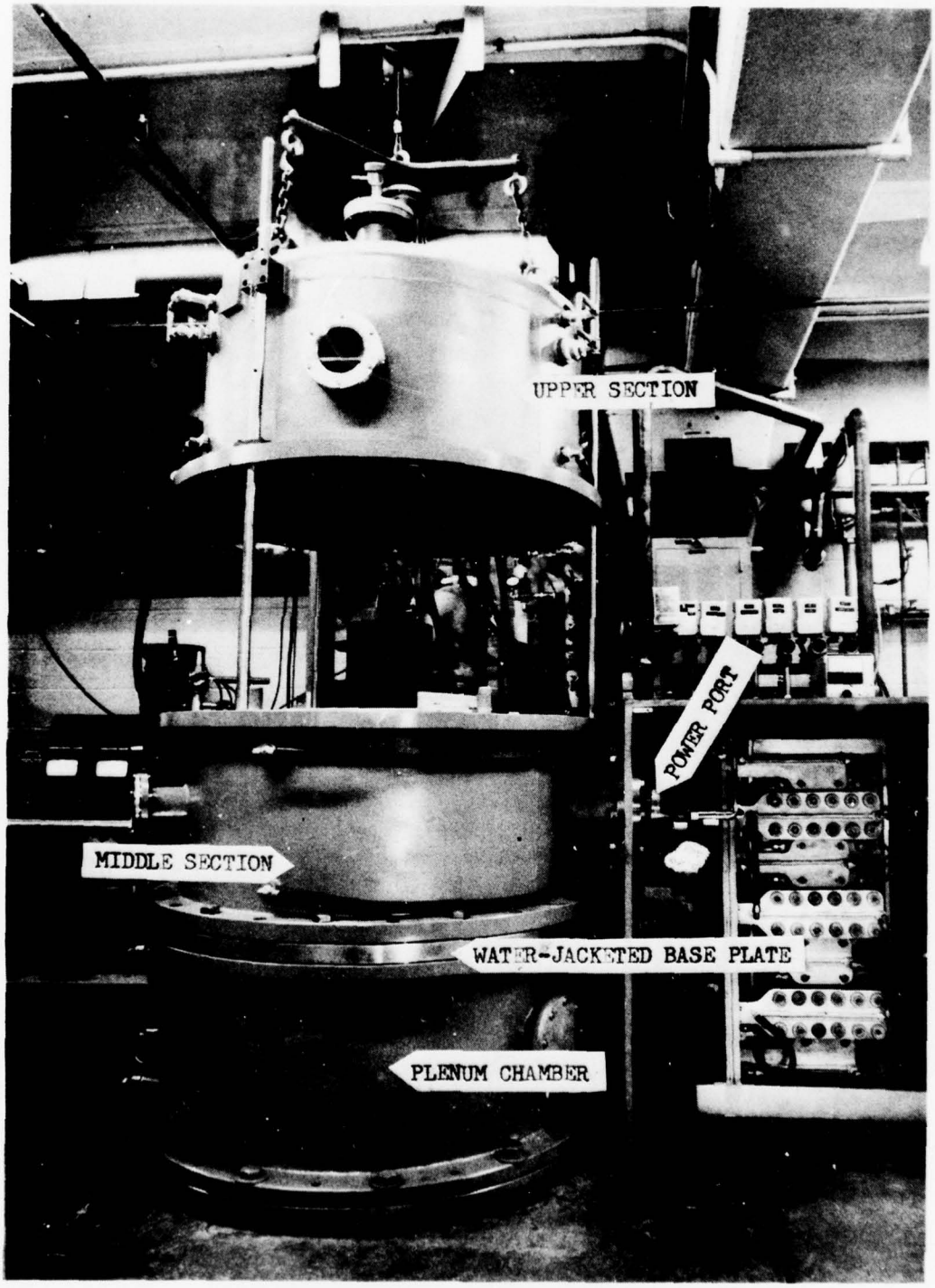

Fig. A-1. Coating Furnace

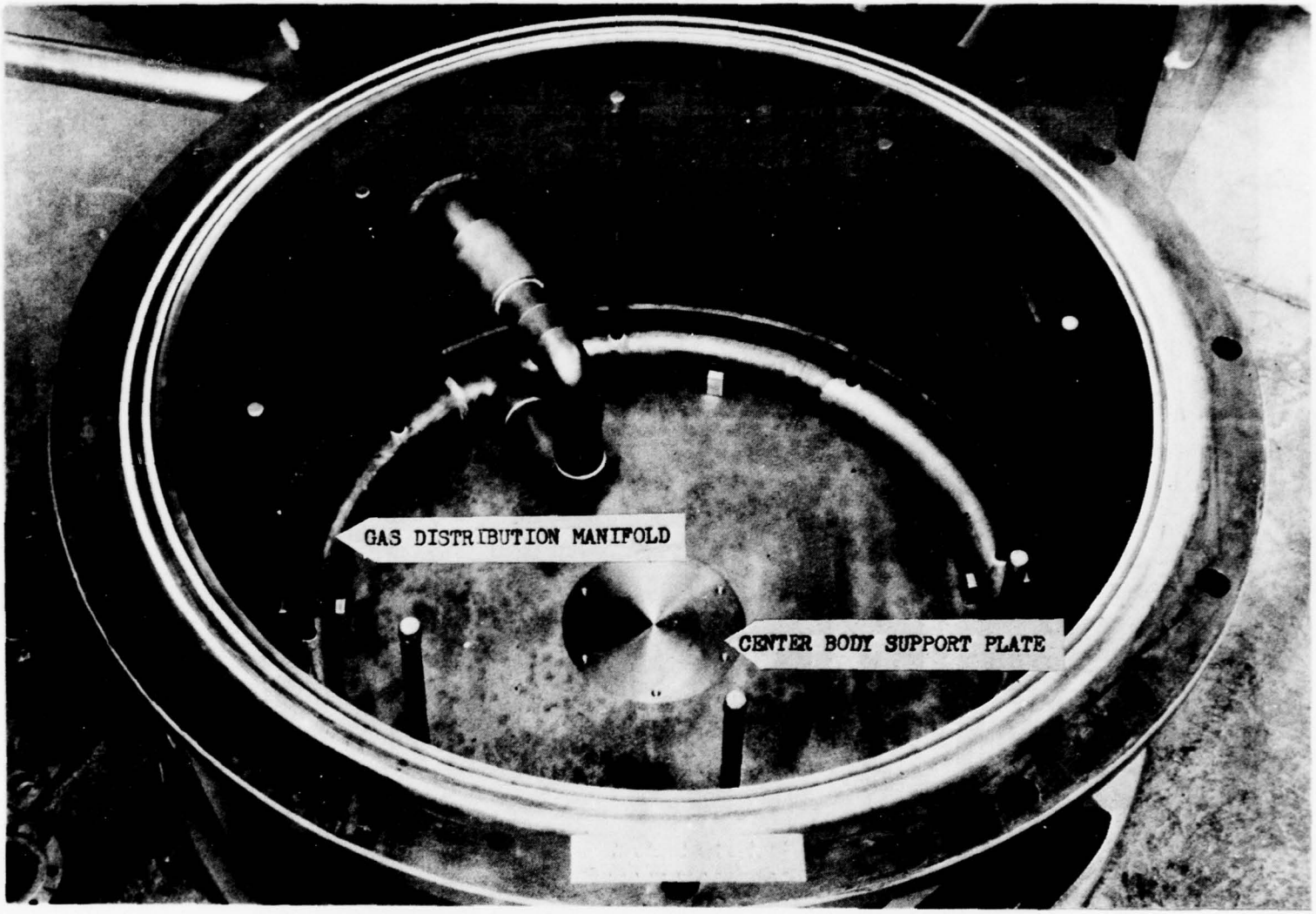

Fig. A-2. Interior view of plenum chamber.

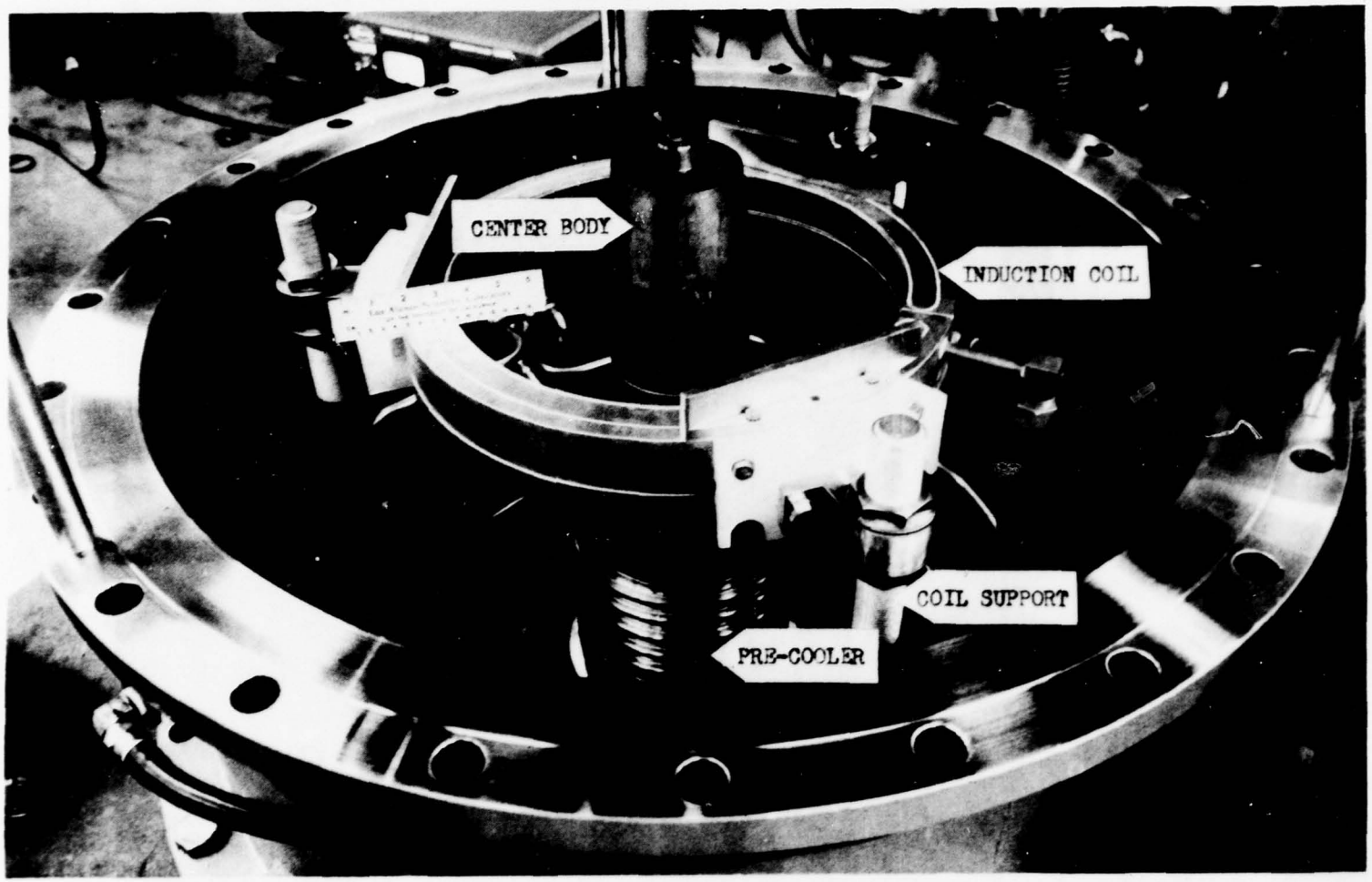

Fig. A-3. Interior view of middle section showing the center body, induction coil and precooler.

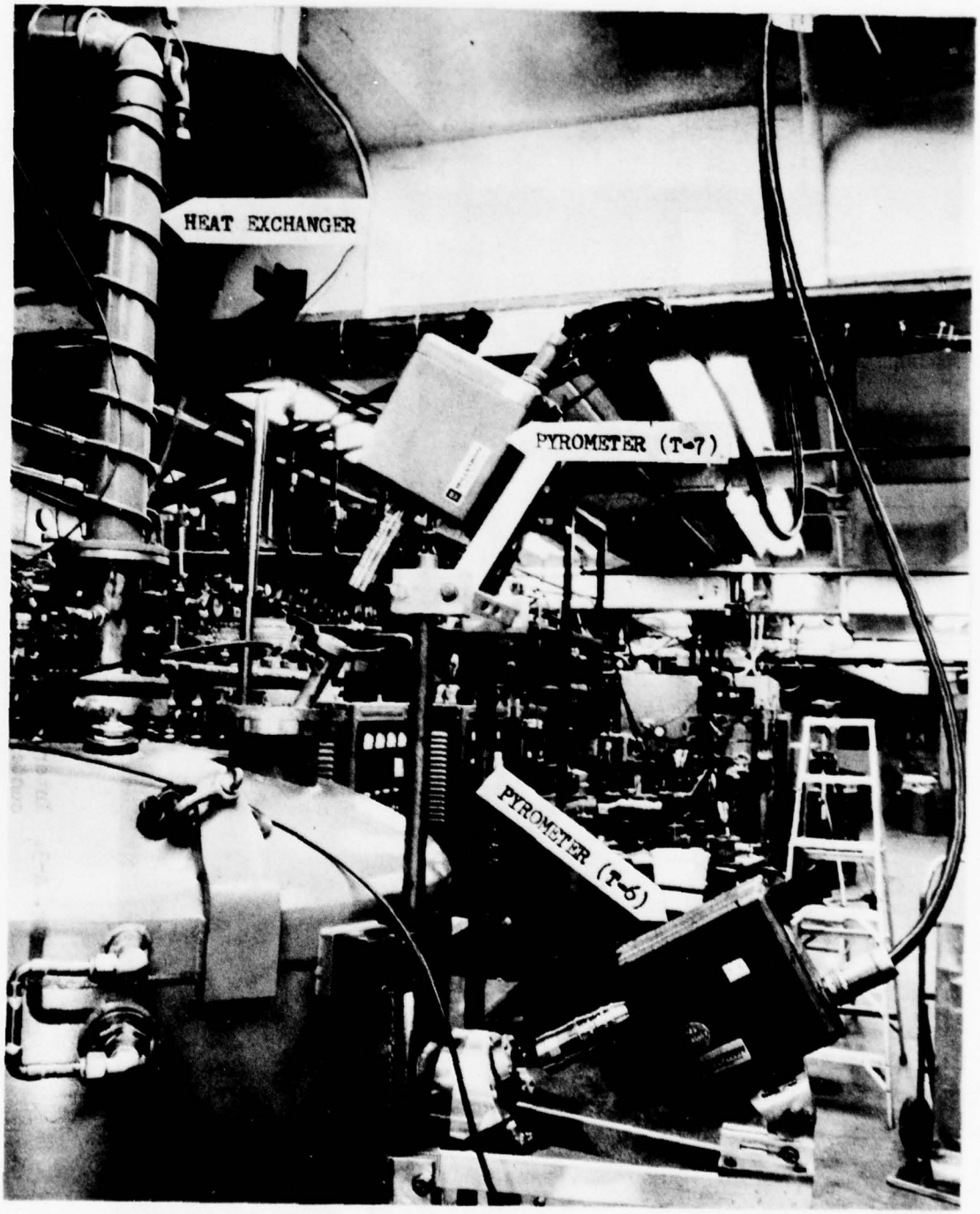

Fig. A-4. Coating system exhaust duct and heat exchanger.

distribution manifold in the plenum chamber.

B. Coating gas constituents - MTS and CH_4.

Coating gas constituents were admitted to the process nitrogen line ahead of the gas distribution manifold in the plenum chamber through a mass flow rate control system. The bottle manifolds with pressure-reducing regulator valves were installed for CH_4 and He. A ventilated enclosure was provided for the CH_4 supply. Purge nitrogen for the Tylan system and the bell jar pyrometer ports was provided from the house nitrogen supply. Compressed air for gas-operated valves in the Tylan unit was piped from the house air system.

C. Cooling Water.

Cooling water for the bell jar jacket, center body, coil support and induction coil, exhaust canopy, exhaust heat exchanger, and capacitor bank was provided by the house circulating water system. Instrumentation to measure flow rates and supply and discharge temperatures was installed.

III. ASSEMBLY

Figure A-5 shows the graphite components of the furnace assembly before installation, and Fig. A-6 shows them installed. Actually the coil was removed from the coil support during assembly to permit wrapping the carbon felt insulation and installing lower thermocouples. The coil was then installed and connected. Figure A-7 shows the complete furnace assembly wrapped in insulation with the thermocouples installed just before the bell jar was closed. The upper bell jar section was then lowered, the exhaust line was connected to the heat exchanger, and the pyrometer mounts were installed.

Detailed parts and assembly drawings are available as Los Alamos Scientific Laboratory, "Co-Deposition Coating Furnace, CMB-3," Drawing No. 26Y-19915, sheets 1-13 (November 1976).

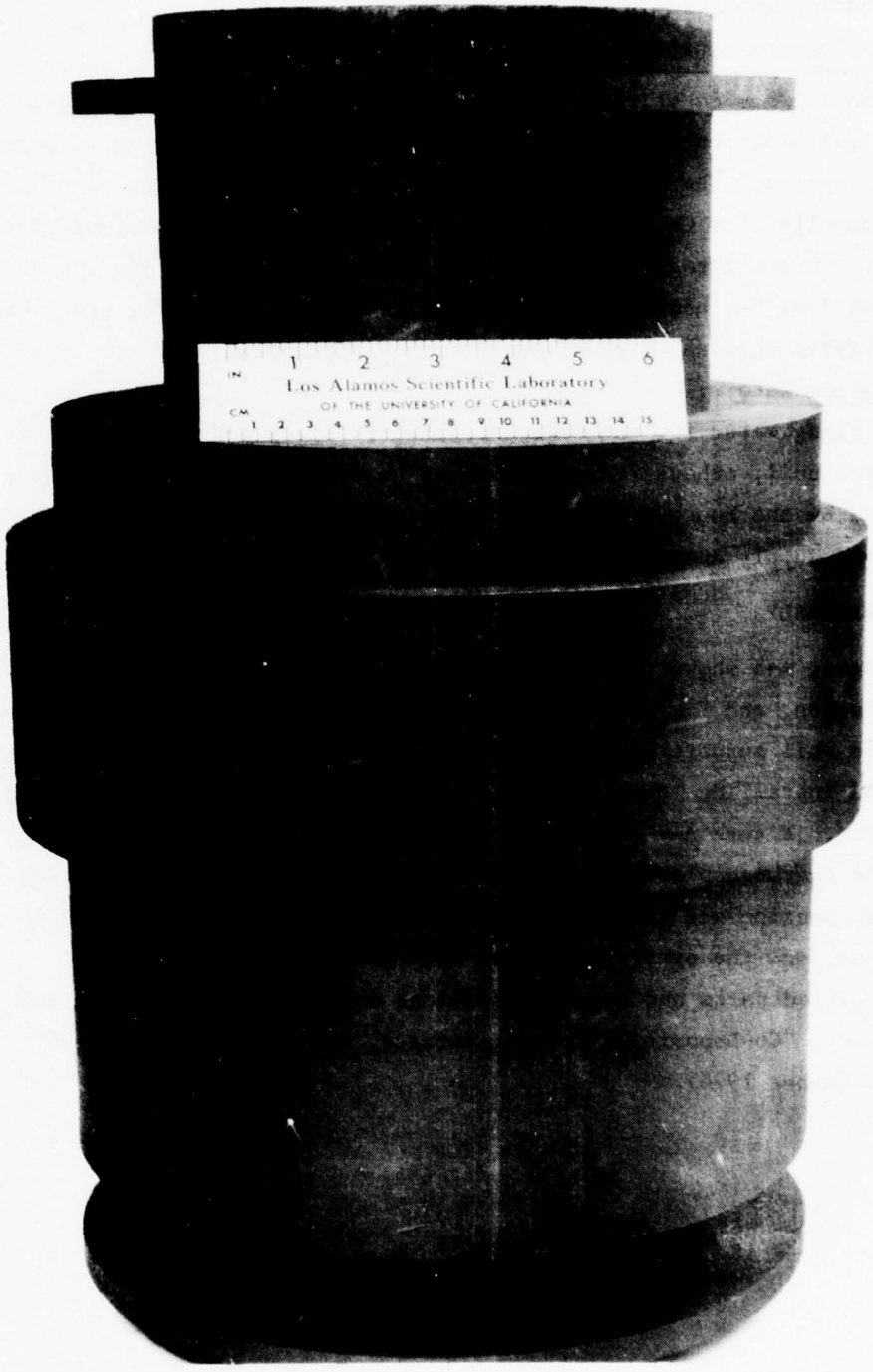

Fig. A-5. Graphite components of furnace assembly.

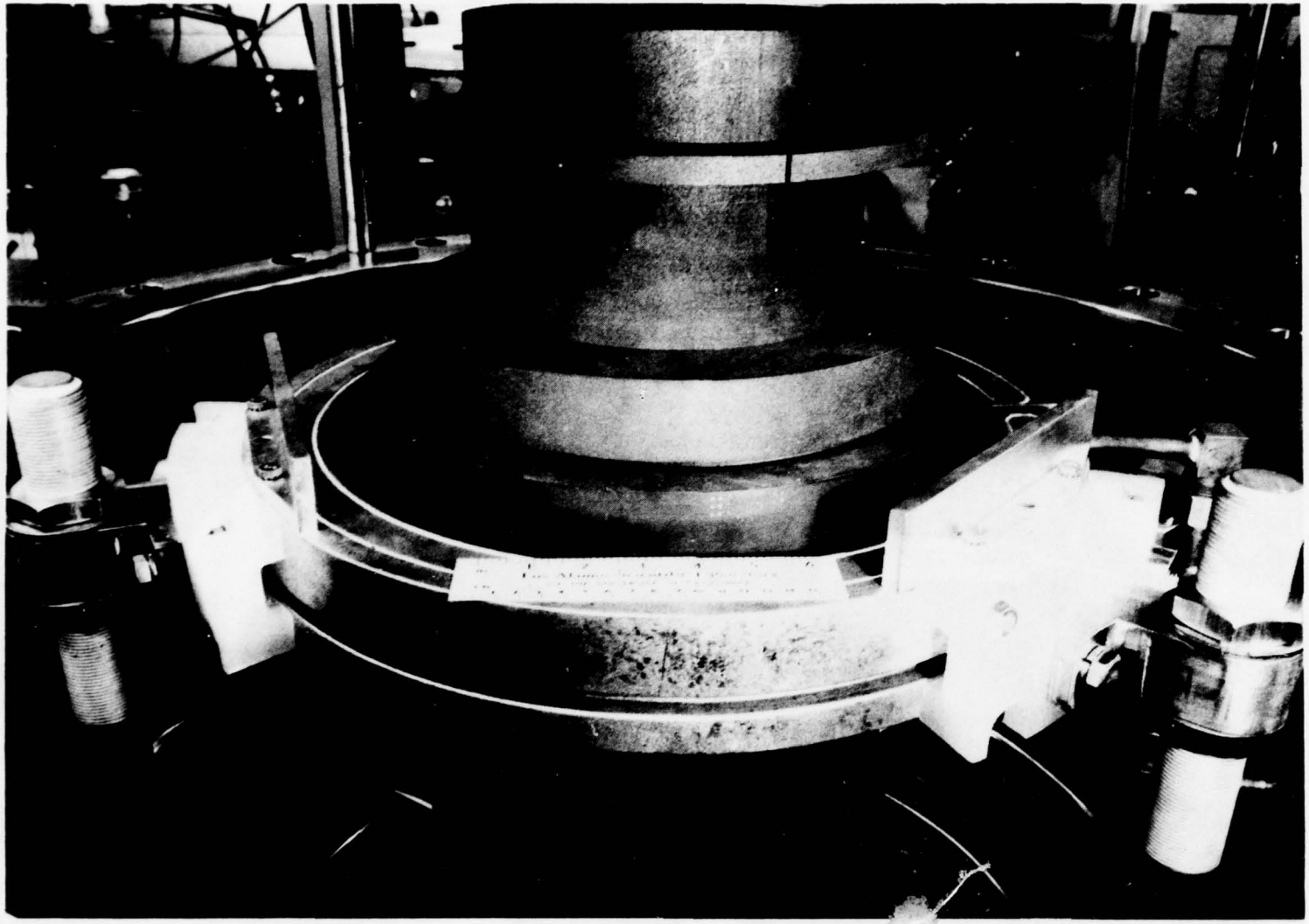

Fig. A-6. Graphite components installed in furnace assembly.

Fig. A-7. Graphite components of furnace assembly wrapped with insulation

APPENDIX B

INSTRUMENTATION REQUIREMENTS AND DESCRIPTION

I. REQUIREMENTS

The data acquisition system (DAS) measured and recorded approximately 33 channels of analog data. The end-to-end channel inaccuracy was to be ±2% maximum. The frequency response of each parameter was essentially dc, and the resolution had to be at least one part in 1000. The Measurement List, Table B-I shows the parameters to be measured, their ranges, and the transducer or method used for the measurement.

II DESCRIPTION

A. General

The DAS consisted of a data logger that multiplexed the 33 analog channels and formatted the data. These data were then recorded on magnetic tape by a seven-track incremental tape recorder. The parameters recorded were 21 temperature measurements, one pressure measurement, 13 flow measurements, two power measurements, and the time of day. Figure B-1 shows the measurement sites on the furnace. The DAS block diagram is shown on Fig. B-2. The instrumented furnace and the DAS are shown in Figs. B-3 and B-4, respectively.

B. Transducer Installations and Signal-Conditioning Section

1. Pressure Measurements. The single static pressure measurement was obtained using a conventional bonded strain-gauge type pressure transducer. The parameter P-1 was measured at the wall of the furnace inlet chamber. Its output was Vdc for 200 kPa full-scale input. The output was connected to a voltage divided to reduce the 5V to 3V to be compatible with the 3V full-scale range of the data logger. The transducer installation is shown in Fig. B-5.

2. Temperature Measurements. Temperature was measured using copper vs Constantan (Type-T) and W-5% Re vs W-26% Re thermocouples (TCs) and two-color type pyrometers.

The low temperature measurements (100-500 K) were made using type-T Tcs with ungrounded junctions. The TCs were 3mm in diameter with a stainless steel sheath. Parameters measured using this Tc configuration were T-1 and T-11-T-21. Fig. B-6 shows a typical installation of these TCs. The units were inserted into a tee in the line so that the TC junction was placed in the middle of the

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TABLE B-1

MEASUREMENT LIST

PARAMETER	PARAMETER DESCRIPTION	RANGE	RESOLUTION	ACCURACY	TRANSDUCER TYPE	TRANSDUCER MANUFACTURER AND PART NUMBER	S/N
F-1	Mass Flow Rate; Process H_2	0 to 1416 SLPM	±0.1 SLPM	±2%	Thermal	Hastings Radist ANL-500(L-50F 50 M/4-5M	361
F-2	Mass Flow Rate; MFS	0 to 9 gpm	±0.00[illegible] gpm	±4%	Thermal	Tylan GF-348	-
F-3	Mass Flow Rate; CH_4	0 to 10,000 SCCM	±5 SCCM	±2%	Thermal	Tylan GF-348	-
F-4	Mass Flow Rate; Ar	0 to 6 SLPM	±0.00[illegible] SLPM	±2%	Thermal	Tylan GF-348	-
F-5	Flow Rate Ratio; MFS/Ar	0 to 30%	±0.01%	---	Calculated	Tylan GF-348	-
F-6	Flow Rate; Cooling H_2O, Bell Jackets	0 to 340.6 SLPM	±0.01 SLPM	±1%	Turbine	Flow Technology FT-20[illegible]C-LA	20034
F-7	Flow Rate; Cooling H_2O, Center Body	0 to 47.3 SLPM	±0.00[illegible] SLPM	±1%	Turbine	Cox Instruments AN-10	24811
F-8	Flow Rate; Cooling H_2O, Heating Coil	0 to 47.3 SLPM	"	±1%	Turbine	Cox Instruments AN-10	24813
F-9*	Flow Rate; Cooling H_2O, Coil Support	0 to 47.3 SLPM	"	±1%	Turbine	Cox Instruments AN-10	24811
F-10*	Flow Rate; Cooling H_2O, Bell Jar Base	0 to 47.3 SLPM	"	±1%	Turbine	Cox Instruments AN-10	24811
F-11*	Flow Rate; Cooling H_2O, Precooler	0 to 47.3 SLPM	"	±1%	Turbine	Cox Instruments AN-10	24812
F-12*	Flow Rate; Cooling H_2O, Top Canopy	0 to 47.3 SLPM	"	±1%	Turbine	Cox Instruments AN-10	24812
P-1	Pressure; Static, Inlet Chamber	0 to 30 PSIA	±0.01 PSIA	±1.5%	Bonded Strain Gage	Standard Controls 212-25-010-13	30869
T-1	Temp.; Gas, Furnace Inlet	100 to 500 K	±0.06 K	±2%	Type T T/C	Thermo Electric, T180-304-0-12-0L	---
T-2	Temp.; Within wall of Inlet Tube (Bottom)	500 to 2000 K	±0.1 K	"	W/ARe T/C	ARI T50386-12-30	---
T-3	Temp.; Within wall of Inlet Tube (Middle)	500 to 2000 K	±0.1 K	"	W/ARe T/C	ARI T50386-12-30	---
T-4	Temp.; Within wall of Inlet Tube (Top)	500 to 2500 K	±0.1 K	±2%	W/ARe T/C	ARI T50386-12-30	---
T-5	Temp.; Carbon Felt, Outside Surface	500 to 1500 K	±0.1 K	±2%	W/ARe T/C	ARI T50386-9-33	---
T-6	Temp.; Substrate, Backside Surface	1300 to 2500 K	"	"	Optical Pyrometer	Millitron, Thera-O-Scope	322
T-7	Temp.; Substrate, Deposition Surface	1300 to 2500 K	"	"	Optical Pyrometer	Millitron, Thera-O-Scope	463
T-8	Temp.; Within wall of Exit Tube (Bottom)	500 to 2500 K	±0.1 K	±2%	W/ARe T/C	ARI T50386-12-30	---
T-9	Temp.; Within wall of Exit Tube (180° from T-8)	500 to 2500 K	±0.1 K	±2%	W/ARe T/C	ARI T50386-12-30	---
T-10	Temp.; Gas, Exhaust	500° to 1500 K	±0.1	"	W/ARe T/C	ARI T-938-12DAE 9X300	---
T-11	Temp.; Cooling H_2O, Supply	273 to 310 K	±0.06 K	"	Type T T/C	Thermo Electric T130-304-0-12-0L	---
T-12	Temp.; Cooling H_2O, Bell Jackets' Discharge	273 to 373 K	"	"	"	Thermo Electric T180-304-0-12-0L	---
T-13	Temp.; Cooling H_2O, Coil Support Discharge	273 to 373 K	"	"	"	Thermo Electric T180-304-0-12-0L	---
T-14	Temp.; Cooling H_2O, Center Body Discharge	273 to 373 K	"	"	"	Thermo Electric T180-304-0-12-0L	---
T-15	Temp.; Cooling H_2O, Bell Jar Base Discharge	273 to 373 K	"	"	"	Thermo Electric T 80-304-0-12-0L	---
T-16	Temp.; Cooling H_2O, Precooler Discharge	273 to 373 K	"	"	"	Thermo Electric T180-304-0-12-0L	---
T-17	Temp.; Cooling H_2O, Heating Coil Discharge	273 to 373 K	"	"	"	Thermo Electric T180-304-0-12-0L	---
T-18	Temp.; Cooling H_2O, Top Canopy Discharge	273 to 373 K	"	"	"	Thermo Electric T180-304-0-12-0L	---
T-19	Temp.; Furnace Room Ambient	273 to 373 K	±0.06 K	±2%	"	Thermo Electric T180-304-0-12-0L	---
T-20	Temp.; Cooling H_2O, Capacitor Bank Discharge	273 to 373 K	±0.06 K	±2%	Type T/C	ARI T-91R-32 FT9C	---
T-21	Temp.; Cooling H_2O, Capacitor Bus Bar Discharge	273 to 373 K	±0.06 K	±2%	Type T T/C	ARI T-91R-32 FT9C	---
W-1	Power; 10 KHz Furnace	0 to 150 KW	±0.1 KW	±1.5%	Transformer	Los Alamos Scientific Lab.	---
W-2	Power; Apparent (EXI)	0 to 150 KW	±0.1 KW	±1.5%	Transformer	Los Alamos Scientific Lab.	---
Time	Time of Day	0 to 365 Days	±1 Sec	±0.1 Sec	DAS Clock	Doric Scientific	---
Ref Ch	DAS Internal Calibration	0.5000 V	±0.0001 V	±0.0001 V	DAS Ref.	Doric Scientific	---

*Not Measured Continuously

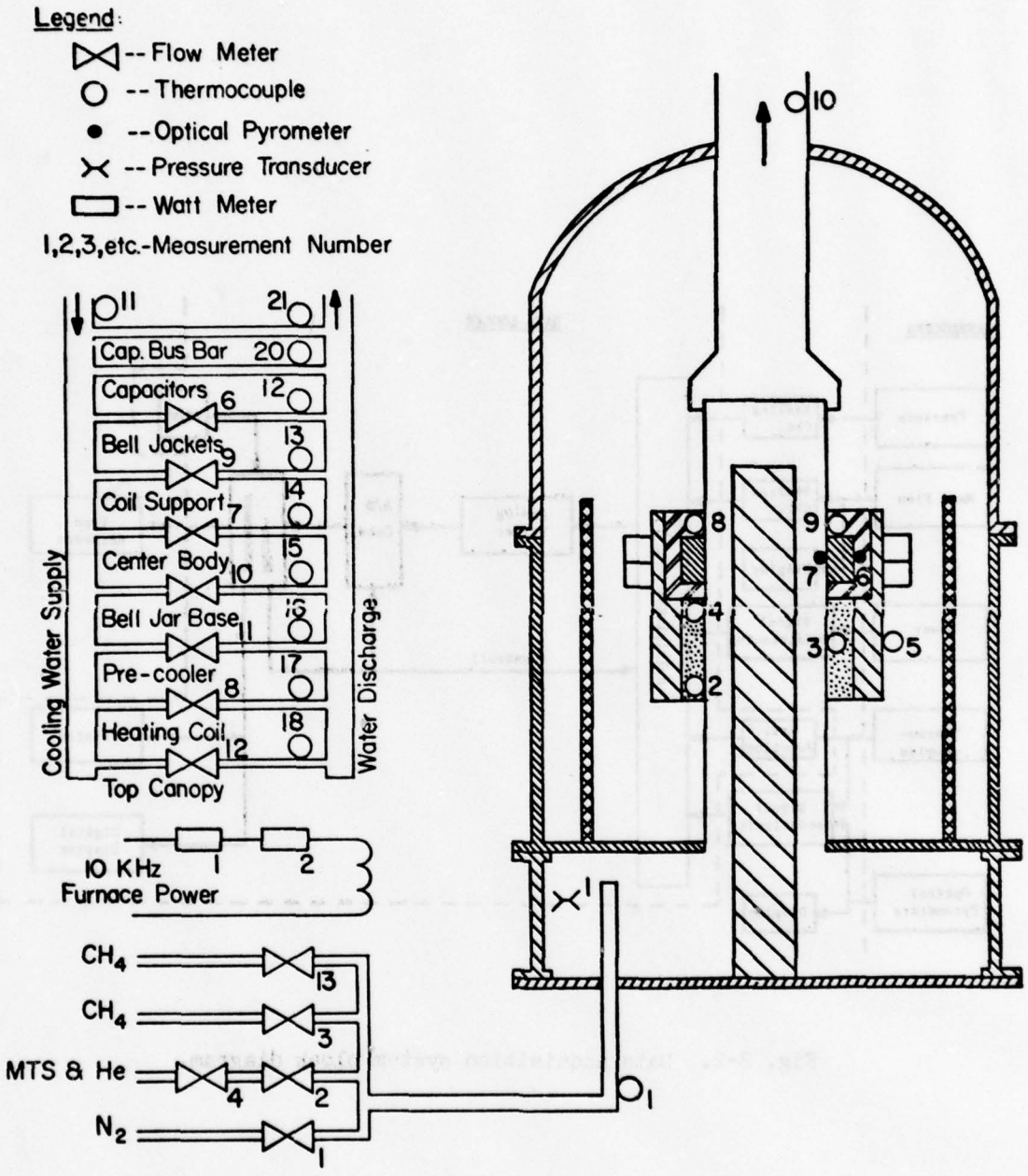

Fig. B-1. Measurement sites.

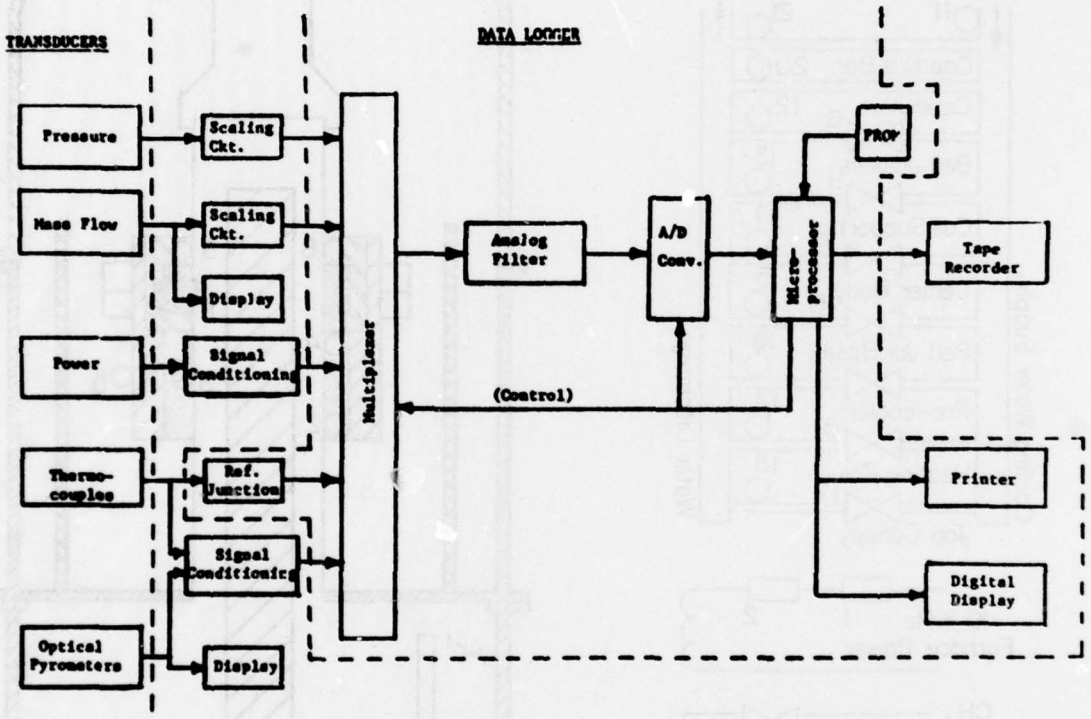

Fig. B-2. Data acquisition system block diagram.

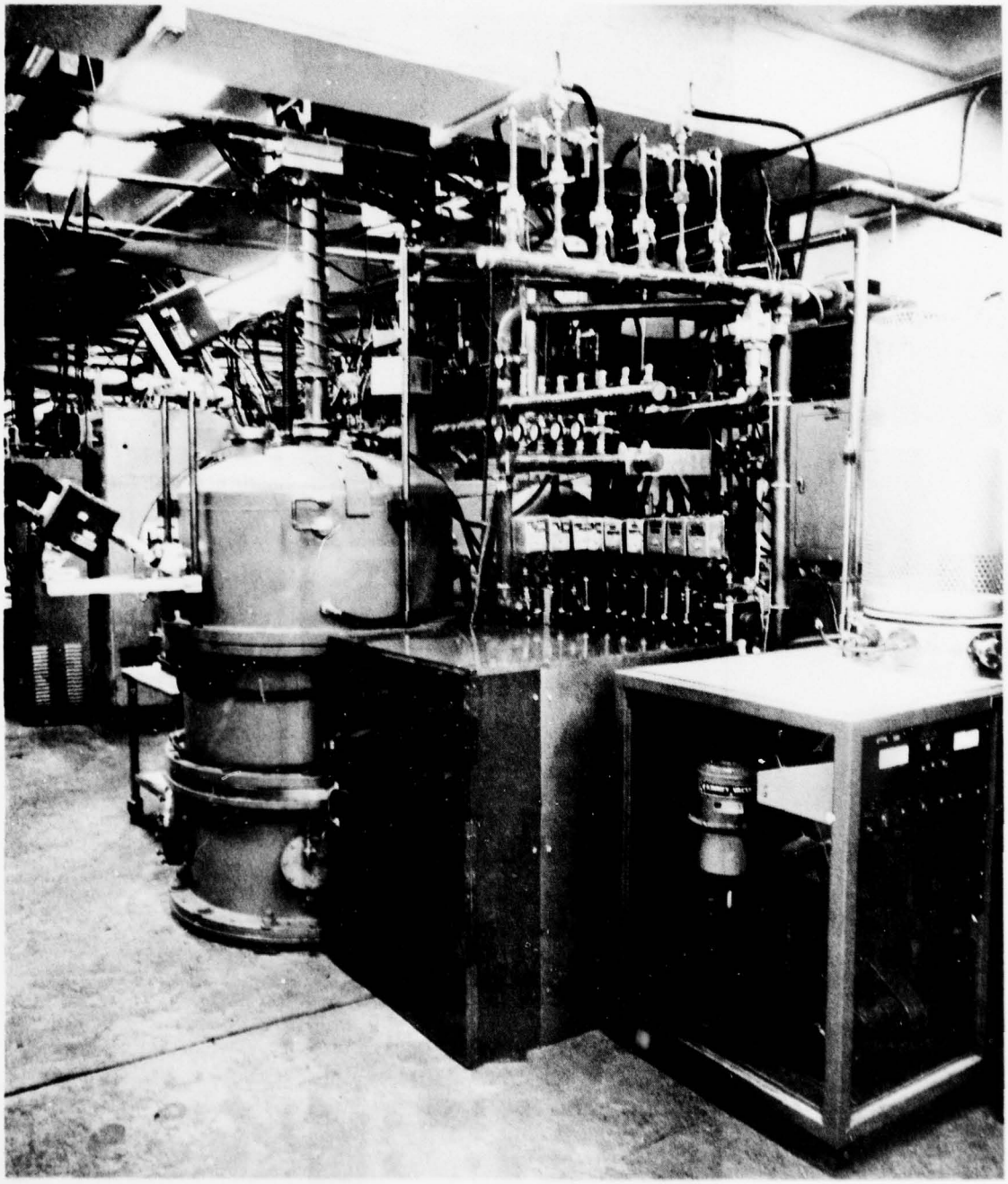

Fig. B-3. Chemical vapor deposition furnace instrumentation.

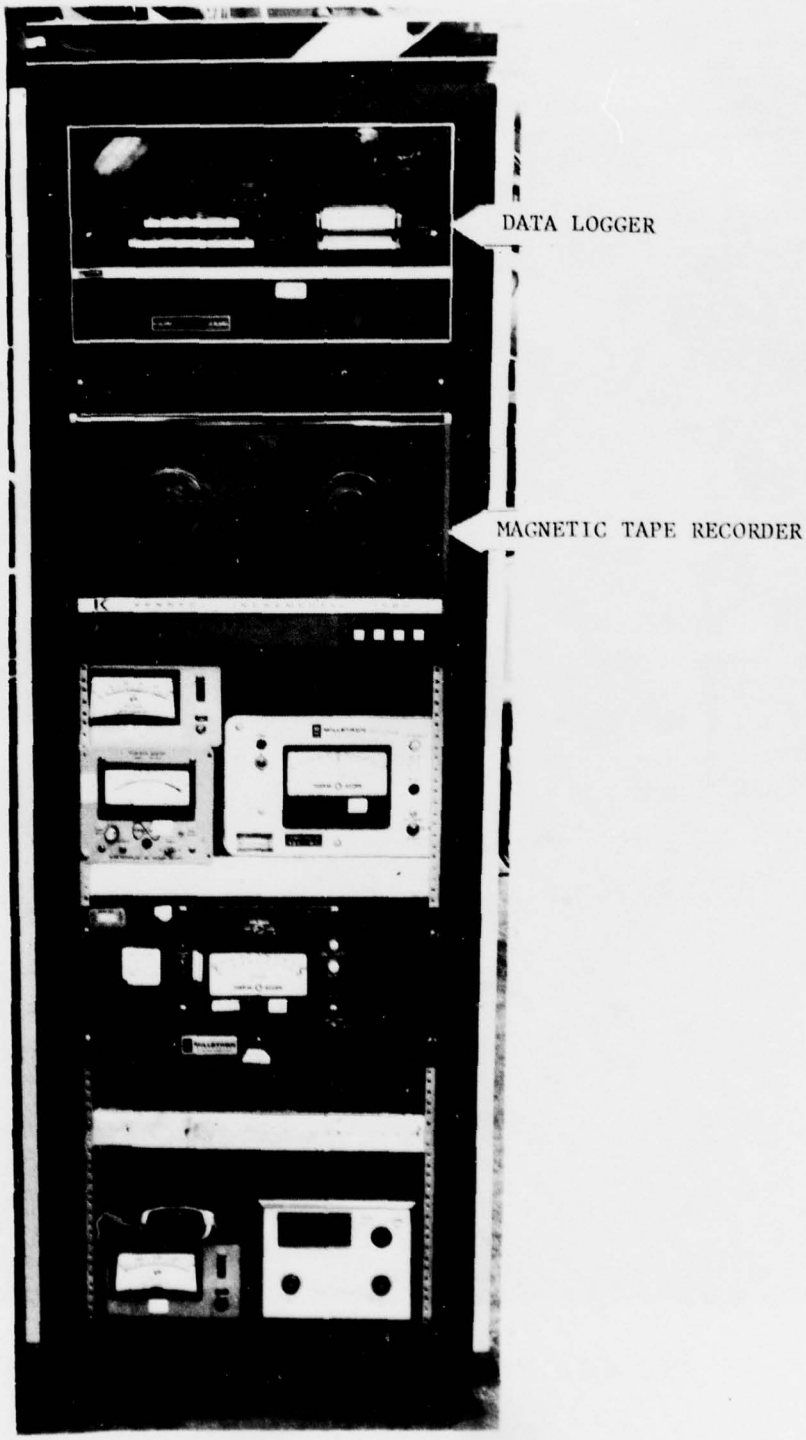

Fig. B-4. Data acquisition system and signal conditioning rack.

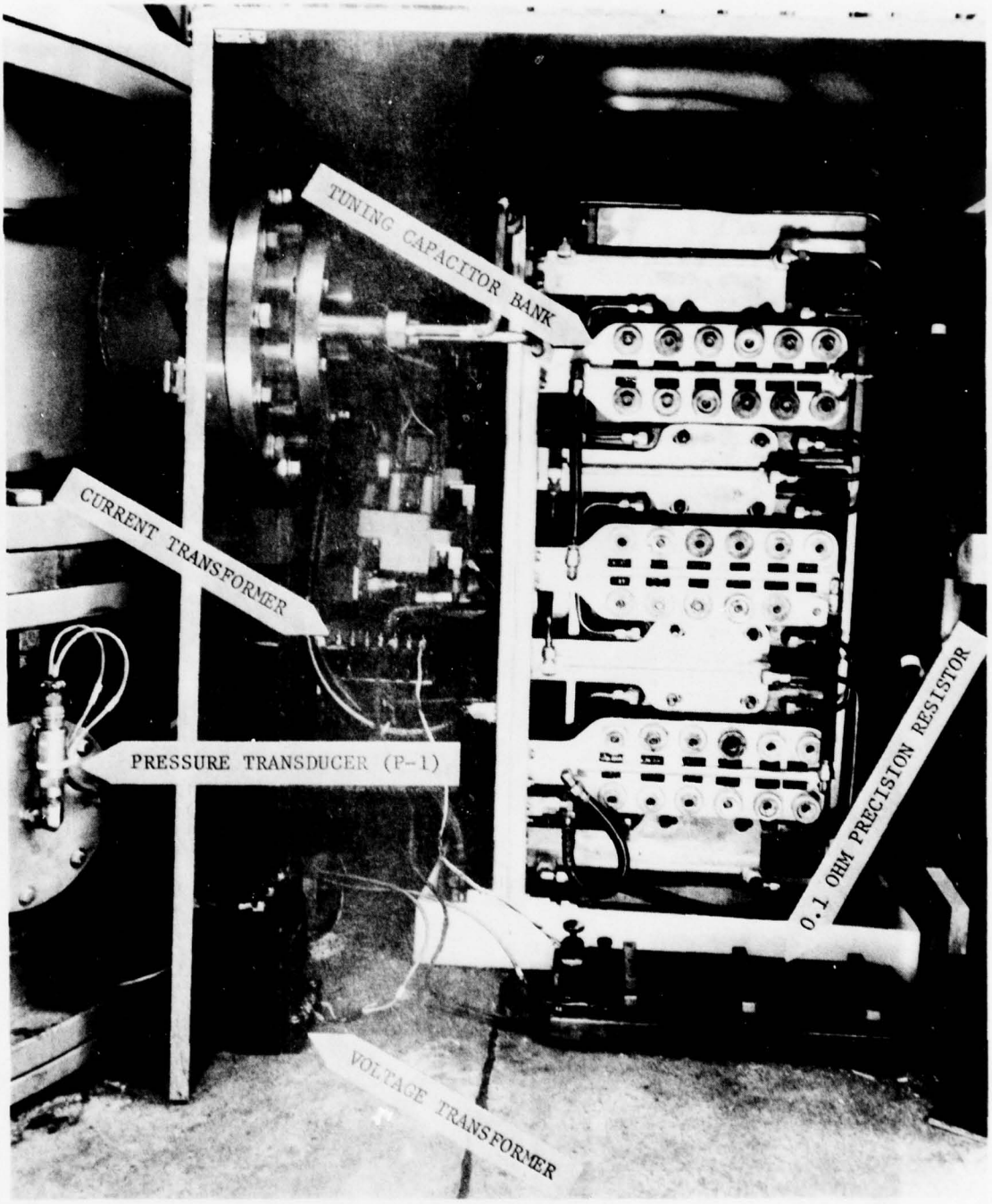

Fig. B-5. Furnace inlet chamber pressure and power measurement.

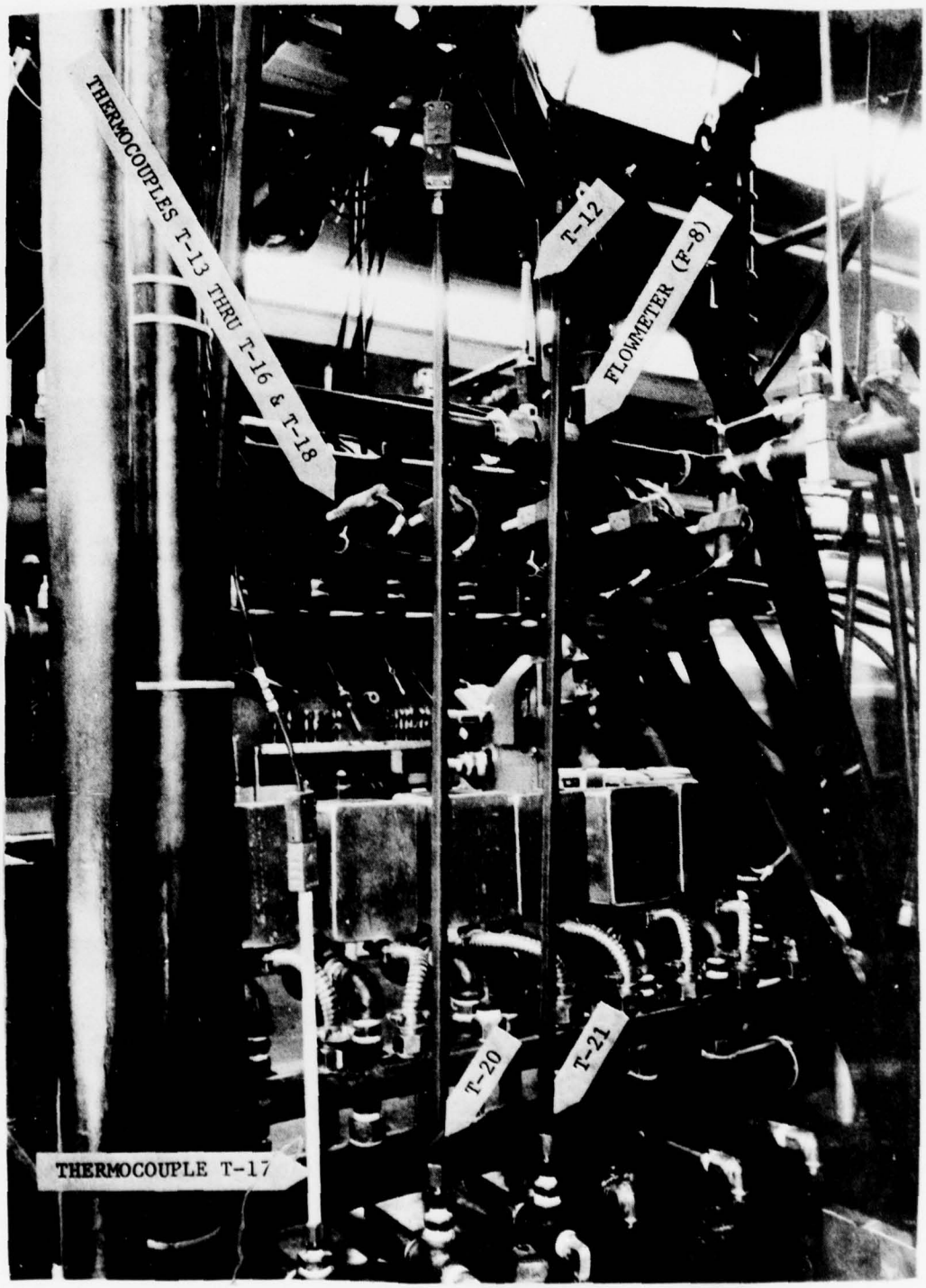

Fig. B-6. Typical type "T" thermocouple installation.

straight section that carried the cooling water. Because all these TCs were excessively long for their application, tubing was cut to length to support the TC projecting out of the tee. It also provided a physical stop to ensure that proper insertion depth was maintained. The strain support for other type-T TCs placed horizontally is also shown in Fig. B-6.

The reference junction for the Type-T TC was contained in the data logger. Premium grade, shielded TC extension wire was used to connect the TCs to the data logger.

Parameters T-2 - T-5 and T-8 - T-10 were measured using ungrounded, W-5% RE vs W-26% Re TCs. The TC sheath was 3-mm-diam. tantalum. Beryllia (BeO) was used for the insulation. Premium grade, shielded TC extension wire was used to connect the units to a room-temperature reference junction. These TCs were wrapped in 28 turns of 0.04-mm thick tantalum foil, and this assembly was inserted into a tantalum thermowell, 7 mm in diameter and 0.4mm thick. The completed assembly was to protect the TC from being contaminated with carbon for ∿100h at 2035 K. Figure B-7 shows a typical thermowell end cap design. The tantalum foil assumes a convex shape at the thermowell tip, thereby presenting a continuous 1-mm cross section of tantalum to the carbon environment.

Figure B-8 shows how the TC sheath ran parallel to the induction coil before leaving the furnace wall through a compression fitting. The carbon substrate and furnace bell jar were both at ground potential, and if the TC sheath contacted the bell jar, and electrical circuit would be completed allowing the current induced by the 10-kHz induction coil to flow in the TC sheath. This current flow at a frequency of 10 kHz would not only have introduced a tremendous amount of noise, but would have provided anomalous data because of TC heating. Therefore, the compression fittings were secured in a 25.4mm thick plastic sheet which, in turn, was used as a pressure seal for the furnace bell jar. This plastic sheet provided an interruption in the electrical circuit to eliminate these error sources.

The TCs for measurement of T-2, T-3, and T-4 were placed 120° apart axially, and they measured the temperature at the bottom, middle, and top of the graphite inlet tube. The TCs were installed in the wall of the graphite tube so that their junctions were 8.9 $\pm$ 1.3mm from the inlet tube's inner surface.

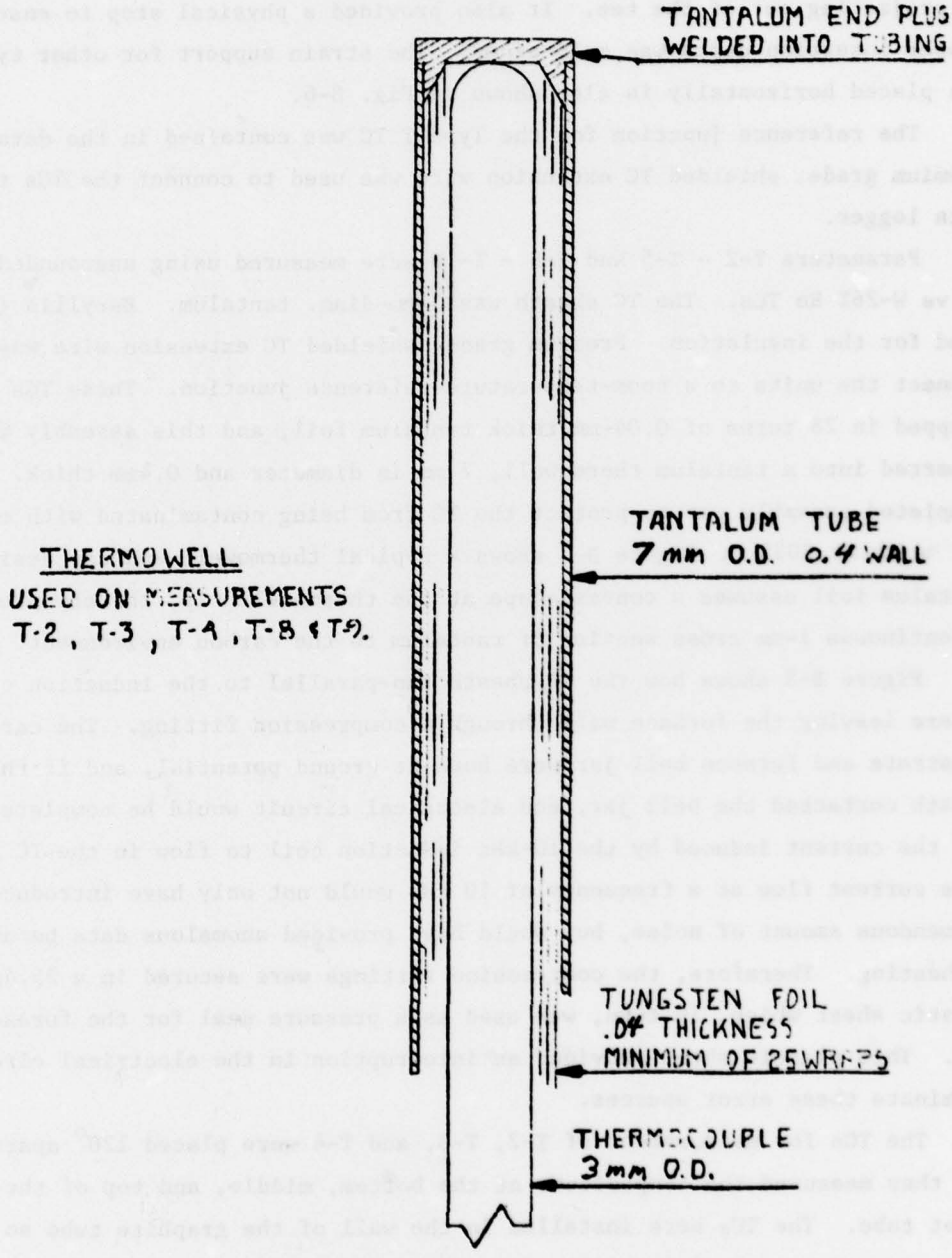

Fig. B-7. Sacrificial thermowell for W/W-Re thermocouples.

Fig. B-8. Typical W-5% Re/W-26% Re thermocouple installation and pyrometer target.

The end of the thermowell for parameter T-5 was placed on the outside surface of the second layer of the insulating felt (second layer from the outside diameter of the assembly). The thermowells for parameters T-8 and T-9 were positioned 180° apart, and their depths were identical to those of the thermowells for parameters T-2, T-3, and T-4. Parameters T-8 and T-9 measured the temperature within the wall of the exit tube. Figures B-8 and B-9 show the installation of the TCs for T-2 and T-8. Figure B-10 shows the installation of the TCs for T-4 and T-9. A typical installation of these TC assemblies into the insulation is shown in Fig. B-8.

The TC that measured T-10 was installed in the furnace exhaust duct. Pressure fittings from an exhaust gas leak. Figure B-11 shows the TC installation. A shield was installed inside the duct to protect the measurement from thermal radiation errors. The TC was inserted inside the 13-mm-diam, stainless steel tube shown in Fig. B-12. During installation, the shield was rotated and locked in place so that the shield slots were placed at approximately 45° to facilitate gas flow through the shield. The signal from the TCs was amplified and filtered by signal-conditioning units to scale the outputs to the full-scale input required by the data logger.

Optical pyrometers were used to measure parameters T-6 and T-7. Each pyrometer "looked" through a clear fused-quartz window and into a 25-mm-i.d. tube to the surface whose temperature was to be measured. The tube was purged constantly with dry nitrogen gas to ensure that the window remained clear. The sight ports for these measurements did not contact the substrate surface, thereby presenting a "closed on one end" sight port configuration, and the substrate, being graphite, was not a pure blackbody. The optical path for measurement of T-6 and T-7 extended through varying proportions of process reactant gases. If there is any radiation-attenuating material in the optical path between the radiant source and the detector, an effect similar to a change in emmitance occurs in the brightness readings of total radiation type pyrometers.

It is theoretically possible to eliminate all of the above errors by using the principle of ratio pyrometry. Here, the ratio of the radiant powers in two wavebands is measured. Wein's Law shows that the ratio of the power in two wavebands emmitted by a heated object is a function of the temperature only. This ratio characterizes the temperature distribution regardless of geometry,

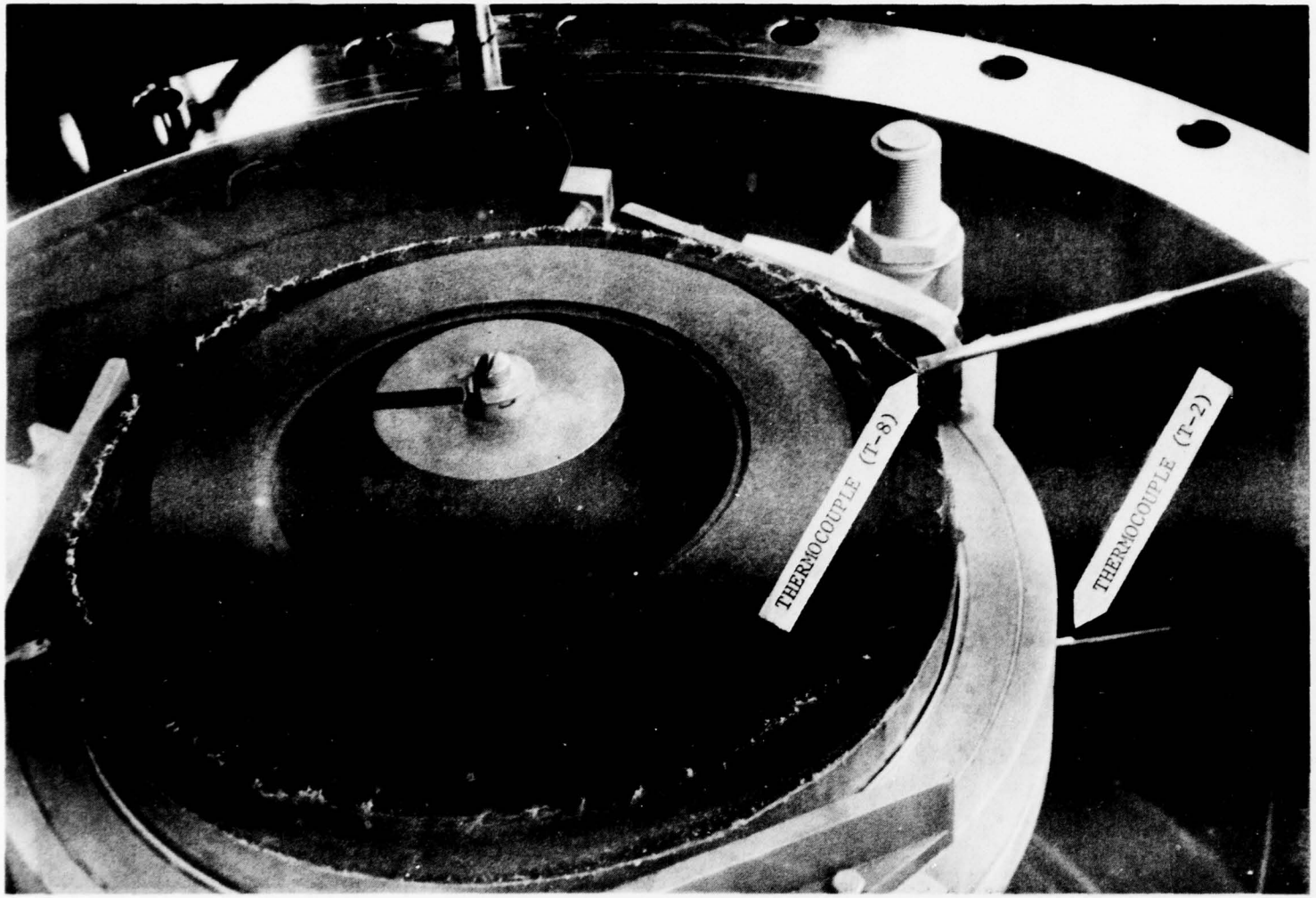

Fig. B-9. Thermocouple temperature measurements of inner graphite wall of furnace.

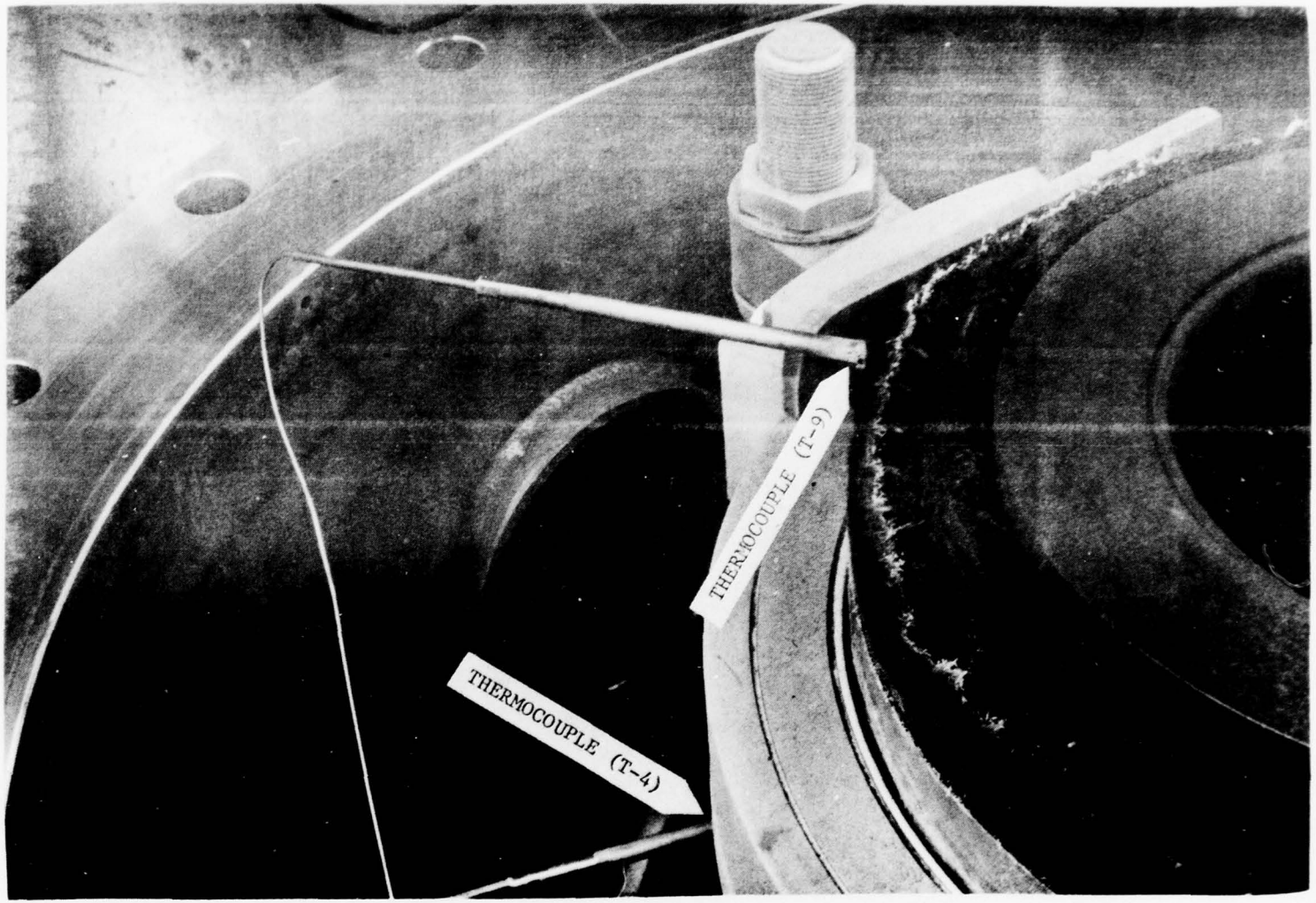

Fig. B-10. Thermocouple temperature measurements of inner graphite wall of furnace.

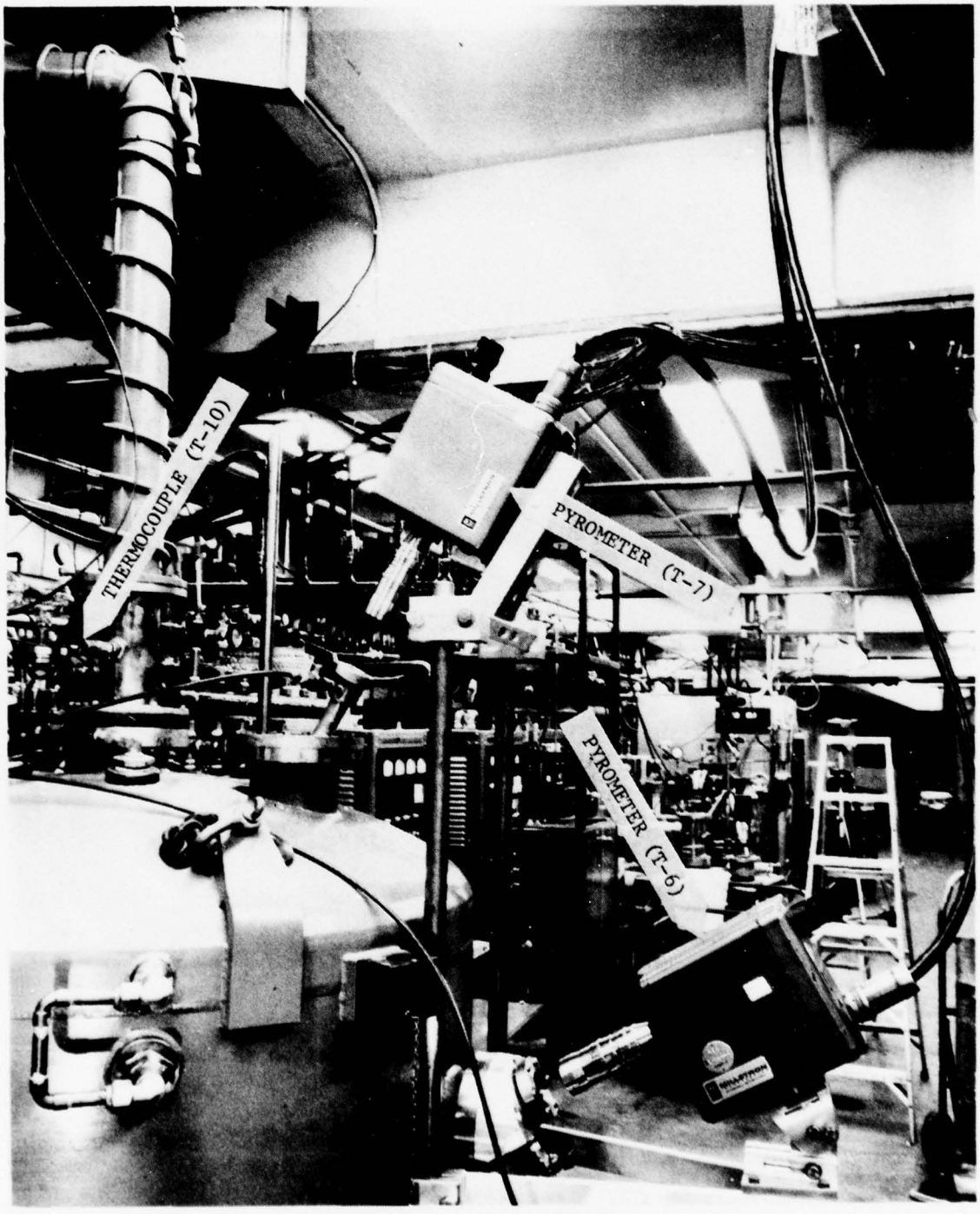

Fig. B-11. Installation of optical pyrometers and exhaust gas thermocouple.

Fig. B-12. Thermocouple thermal radiation shield.

emittance (if the material is a gray body), and transmittance. If the two wavebands are suitably chosen, a linear relationship between the ratio and the temperature, over a wide range of temperatures, can be obtained. Therefore, two-color pyrometers were used to measure the temperature on either side of the substrate to eliminate the requirement for any such corrections.

An error in temperature measurement can be introduced if the optical path is not perpendicular ($\pm 10^{o}$) to the quartz window in the sight ports. This error is associated with reflections from the window, polarization, and attenuation. This source of error was eliminated by mounting the pyrometers within two degrees of perpendicular to the quartz windows.

Another error source could be introduced by not having the pyrometer optics normal ($\pm 10^{o}$) to the substrate surface. To eliminate this source of error for T-6, a spot was machined on the back of the substrate so that the surface was perpendicular to the optical path of the pyrometer. The machined surface of the substrate is shown in Figs. B-8 and B-13. The carbon felt that insulated the entire assembly was cut away in this area to expose the surface to the pyrometer. It is estimated that an error of approximately 10 K would have occurred if the quartz window and the target surface were not perpendicular to the pyrometer optics within the above limits.

3. Flow Measurements. Flow rate and mass flow rates were measured with turbine and thermal-type flowmeters respectively. The parameters cover a range from three g/min to 0.3 m^3/min. All flowmeters were installed so that a length of straight pipe with constant diameter was connected to their inlets and outlets. This straight section was at least five times and three times the flowmeter diameter for the inlet and outlet, respectively. This condition facilitated the velocity distribution requirement to achieve accurate data. All the output signals were 5V full-scale. These signals were reduced to 3V full-scale by a 5:3 voltage divider before being applied to the data logger input.

The parameters F-1 and F-13 were measured with a thermal-type mass flowmeter. These units measured true mass flow without corrections or compensations for the gas temperature and pressure. They operate on the thermal principle that depends on the mass flow of the gas and its heat capacity to change the temperature along a heated conduit. This temperature change is measured by an external arrangement of thermocouples and does not require any sensing elements or projections into the flow stream.

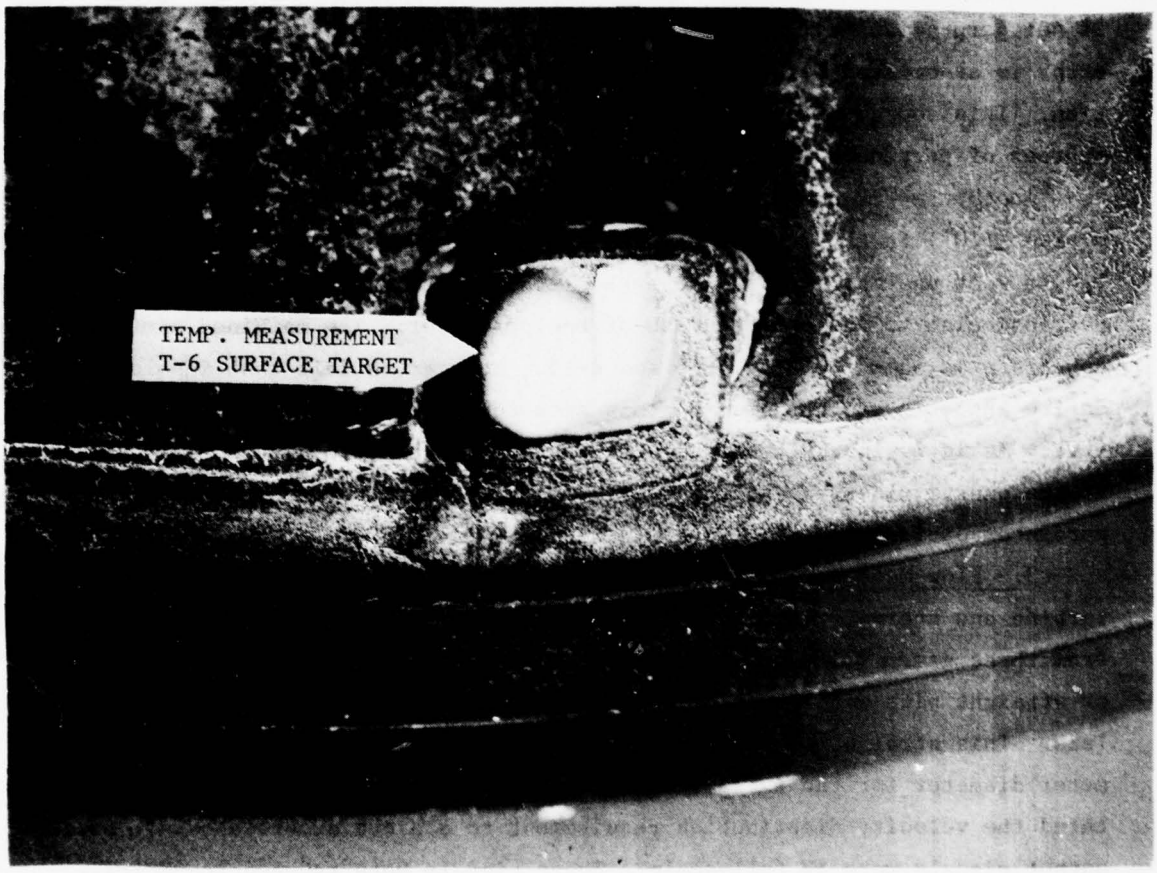

Fig. B-13. Substrate surface preparation for optical pyrometer temperature measurement.

Parameters F-2, F-3, and F-4 were measured using a similar principle. The temperature rise of a gas is a function of the amount of heat added, the mass-flow rate, and gas properties. These mass-flow meters incorporated two resistance-type temperature sensors wound adjacent to each other on the outside of a sensor tube. They formed part of a bridge circuit and had a power dissipation of 40 mw each. When there was no flow in the tube, both sensors were at the same temperature, the birdge was balanced, and the output signal was zero. When there was flow in the tube, the upstream sensor was cooled and the downstream sensor was heated, which produced a signal from the bridge proportional to flow. This signal was then amplified and linearized.

To facilitate accurate control of these parameters, controllers were used to set and regulate their mass flow rate. The CH_4 controller was a standard proportional controller, whereas that for the MTS was unique. The MTS mass was controlled by controlling the mass flow rate of gaseous helium. This was accomplished by injecting helium carrier gas into the bottom of a tank of liquid MTS. A controller using the above thermal principle measured and controlled the helium mass flow rate as a function of the demand for MTS. As the carrier gas bubbled through the liquid MTS, it vaporized the liquid, and and the combined helium and MTS passed through a second thermal flowmeter. This second unit had one self-heated element positioned in a cavity through which the carrier gas flowed and another in a cavity through which the mix flowed. These elements conducted heat through the gases to the base. The temperature difference between the element and the base was proportional to the thermal conductivity of the gas. The thermal conductivity of the mixed gases depended on the ratio of source to carrier.

Parameter T-7 was measured like T-6, except that the substrate was not perpendicular to the optical path of the pyrometer. The angle between the substrate surface and the pyrometer was approximately 50 degrees, resulting in an estimated error of $-5^{\circ}K$. Figure B-14 shows the slot in the water-cooled center body that constituted part of the optical path for measurement of T-7. Figure B-15 shows the sight tube for T-7 as seen by the substrate. Figure B-11 shows the pyrometers for measurements T-6 and T-7. By comparing the temperature of the element on the carrier side to that of the element on the mixture side and by using proper amplification and linearization, we got a 0- to 5-Vdc for zero to full-scale flow ratio. Another circuit electronically multiplied

Fig. B-14. Slotted centerbody showing optical path to substrate for pyrometer T-7.

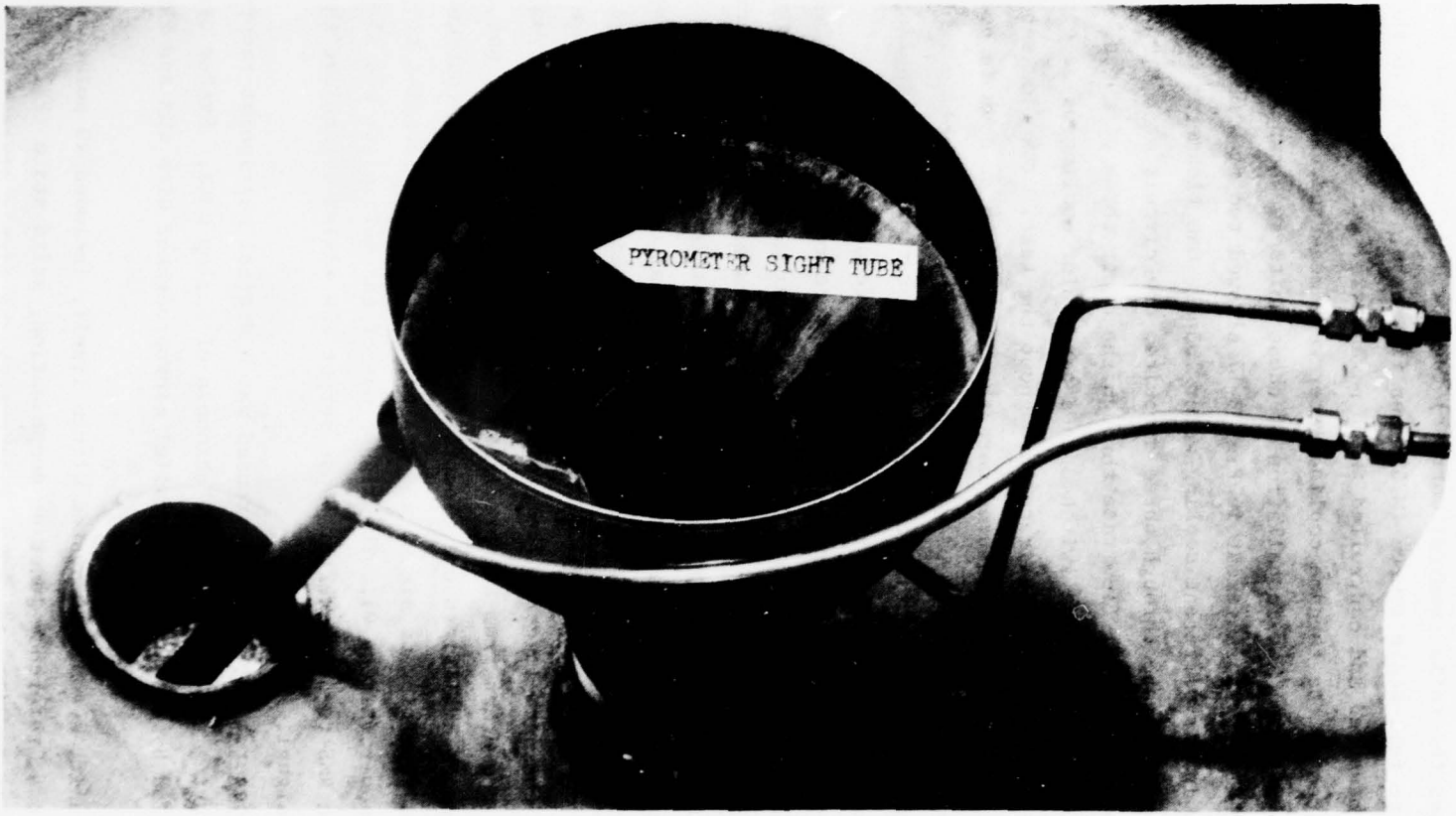

Fig. B-15. Pyrometer Sight Tube Installation.

carrier flow times the ratio (source: carrier) to give a 0- to 5-Vdc source output for zero to full-scale source (MTS) mass flow rate. Figure B-16 shows the system that measured and controlled these parameters.

The flow rates of the furnace cooling water were measured using standard turbine type flowmeters. The installation of these units is shown in Figs. B-6 and B-17. The valve arrangement in Fig. B-17 allowed two flow meters to measure the flow in six different cooling water loops. One flowmeter measured the cooling water flow rate through three circuits, one circuit at a time. The other measured the individual flow rate through the other three circuits. During the test, the two flowmeters are switched to a particular cooling water circuit and continued to measure that flow rate throughout the test. The flow rates of the four circuits not being measured were then ratioed up or down in proportion to any change in flow rate caused by supply pressure fluctuations in the measured circuits. This correction to the basic calibration of the cooling water circuits was accomplished by the computer data reduction program.

4. Power Measurements. Two power parameters were recorded, true power (EI Cos ϕ) and apparent power (EI). The 10 000-H_z voltage and current signals were obtained through isolation voltage and current transformers. The current signal was applied to a 0.1-ohm precision resistor positioned adjacent to the transformer (Fig. B-5) to eliminate errors in the signal caused by transformer loading and an IR drop associated with lead wire resistance. This voltage signal was then applied to a signal conditioner located approximately 30 m away and adjacent to the data logger. The two voltage signals (one from the voltage transformer and the other from the precision resistor) were scaled by use of high input impedance amplifiers and electronically multiplied by a four-quadrant multiplier. The resulting dc signal was scaled, filtered, and applied to the data logger as power (EI Cos ϕ). The signal out of the multiplier was also ac coupled to an RMS-to-dc converter whose output was scaled to achieve the apparent power signal.

5. Data Logger. The data logger used was a digital multipoint recorder, capable of measuring and formatting 40 channels of analog data. System operation is controlled by an eight-bit parallel microprocessor with ROM and PROM microprograms.

The basic unit, a complete system within itself, included 40 points of input terminations, reference junction compensation, solid state (FET) multiplexing, microvolt-level analog-to-digital conversion with a digital display

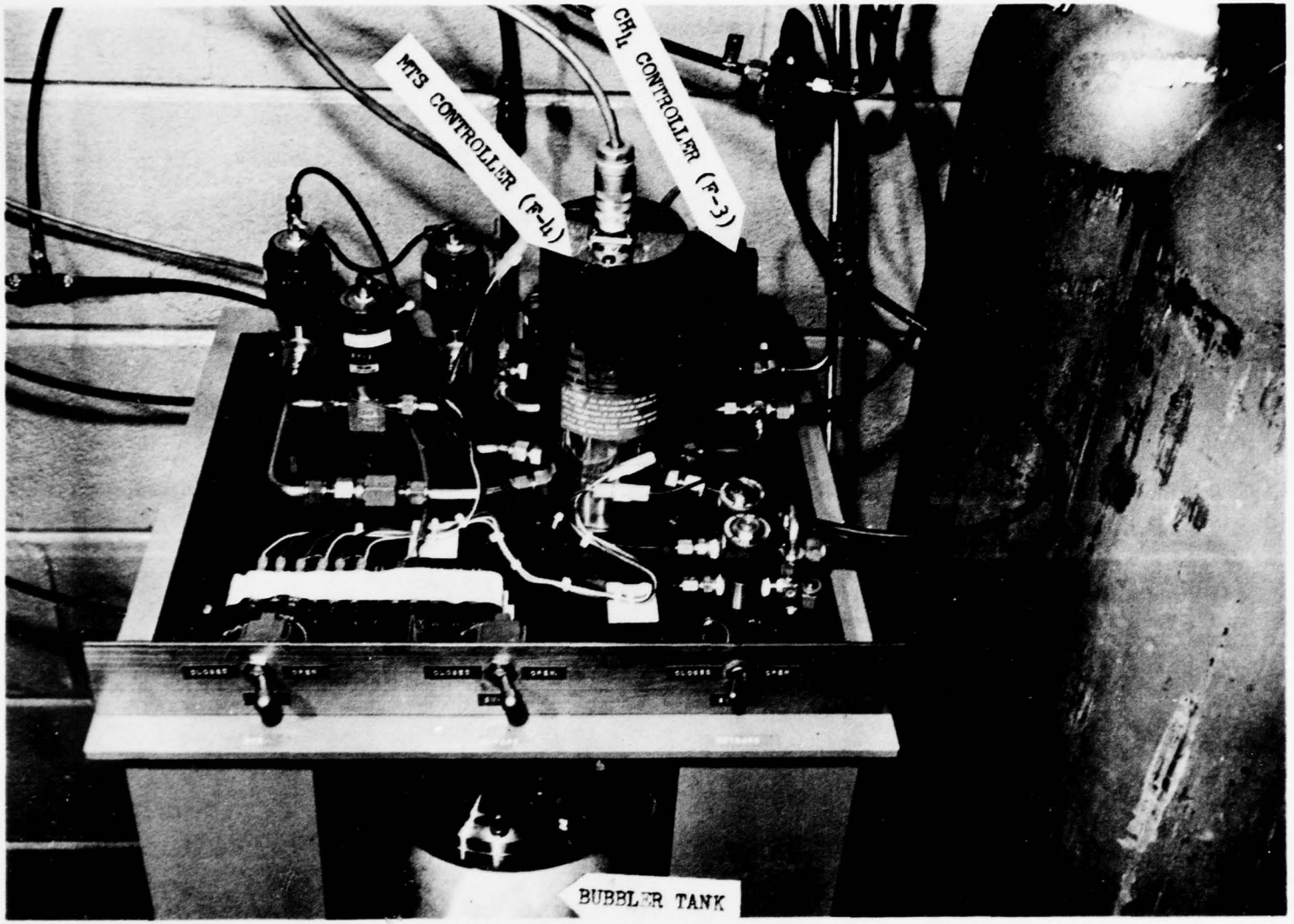

Fig. B-16. Process gas control system.

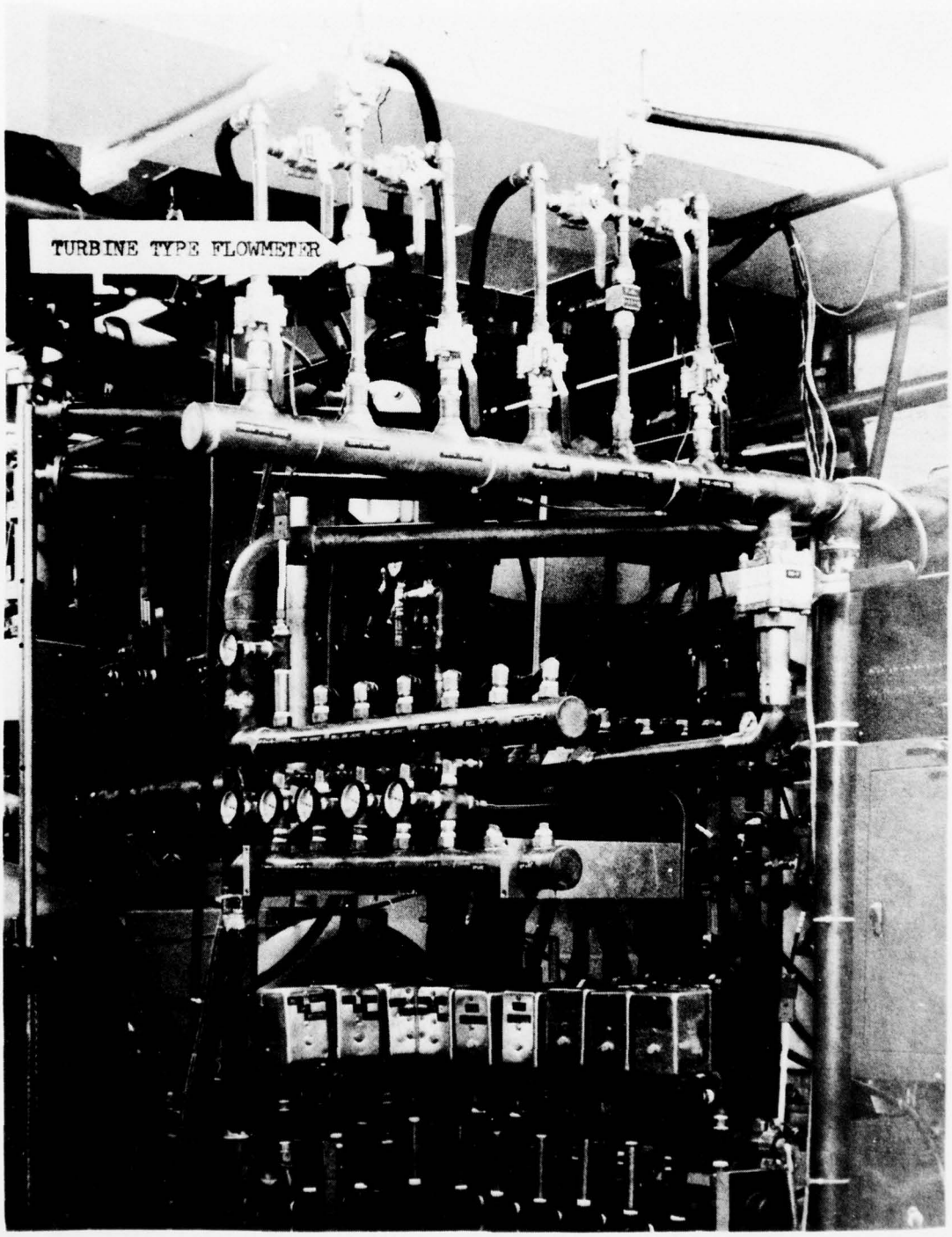

Fig. B-17. Cooling water manifold.

in engineering units, printout on a built-in strip printer, an internal alarm system, and a built-in electronic timer to initiate selective periodic logging cycles for unattended operation.

Front panel controls were provided in the form of pushbuttons and thumbwheel switches, Fig. B-18. The upper series of six pushbuttons operated the time-of-day clock, peripheral devices,the alarm system, and the internal printer. The lower series of eight pushbuttons provided the mode selection and scan interval controls. FIRST POINT and LAST POINT thumbwheel switches, each with three decades, selected the low and high scan points and set the time-of-day clock.

The unit had a full 16-bit digitizer capable of 10- or 100-μV resolution or 0.2 K TC resolution, depending on the range the operator selected. An all-digital method of linearization for near perfect match to NBS TC tables eliminated analog shaping circuit drift.

The digital display provided point address, magnitude, (with negative polarity indication), time-of-day (h, min, and s), and units of measurement readout. In addition, an "I/O" neon light shone to indicate that an error had been detected in a peripheral instrument. The display shone when the power switch was activated and flashed to inform the operator of power on or power interrupt, alarm, I/O failure, or low tape.

An internal circuit card was used to couple the data logger to peripheral equipment such as the seven-track incremental magnetic tape recorder.

The output of this card includes the digitized data, an operator-selected test number, time-of-day (d, h, min, and s), and peripheral control signals.

The internal printer had a typical recording rate of two points per s. It printed out the same information that was provided to the other peripheral equipment.

6. Magnetic Tape Recorder. The digital data from the data logger were recorded on magnetic tape by the incremental magnetic tape recorder. The unit used half-inch-wide tape and recorded in an IBM seven-channel BCD code. In a seven-track system, six of the tracks are data channels and the seventh is the parity channel. Even parity was used for the BCD coding.

7. Gas Analyzer. To better understand the deposition process kinetics, we used a gas analyzer to examine the properties of the exhaust gas during the tests. It was installed as shown in Figs. B-19 and B-20. Figure B-20 shows the connections for the two stages of vacuum pumping used.

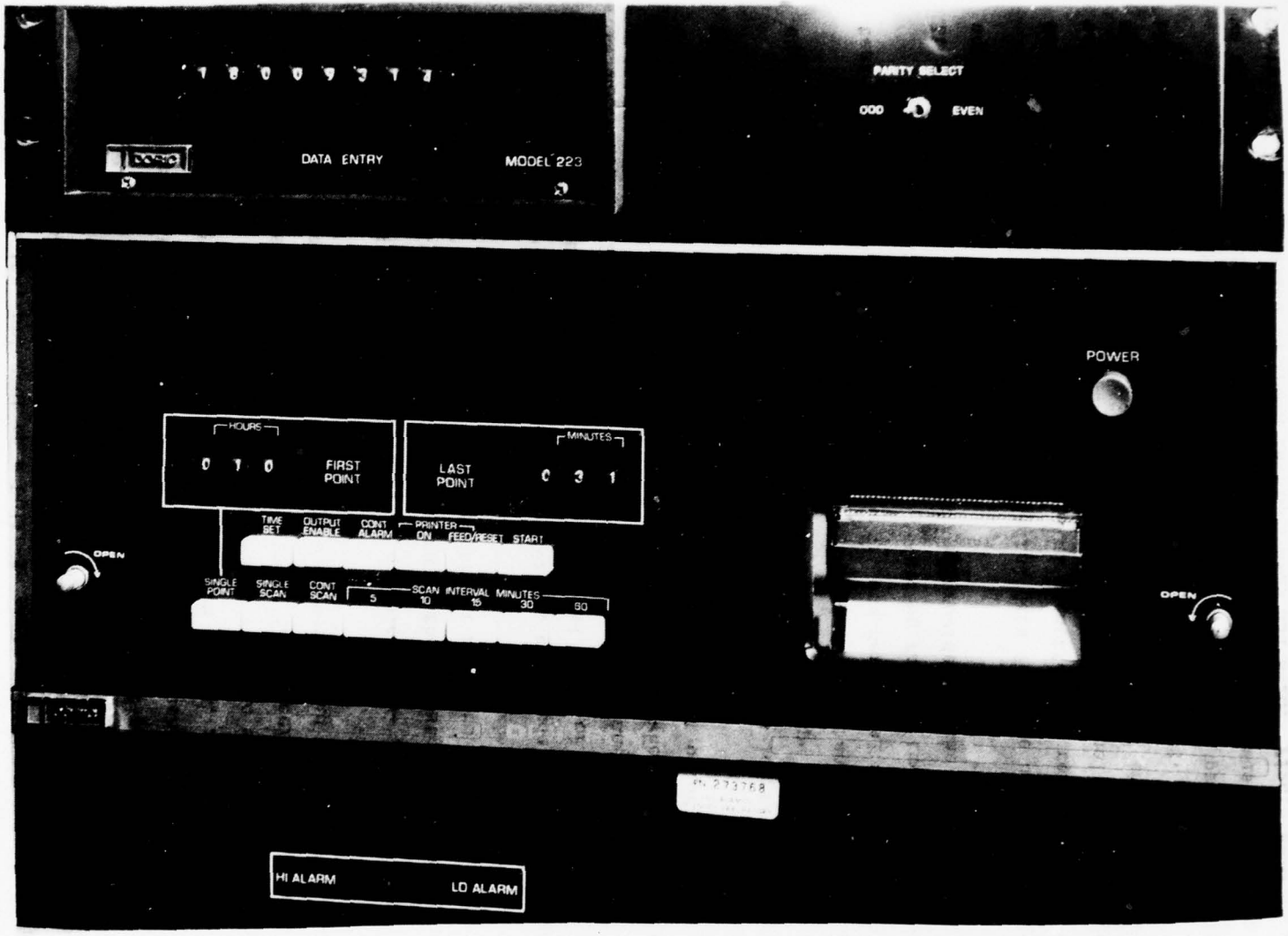

Fig. B-18. Data acquisition system Data Logger.

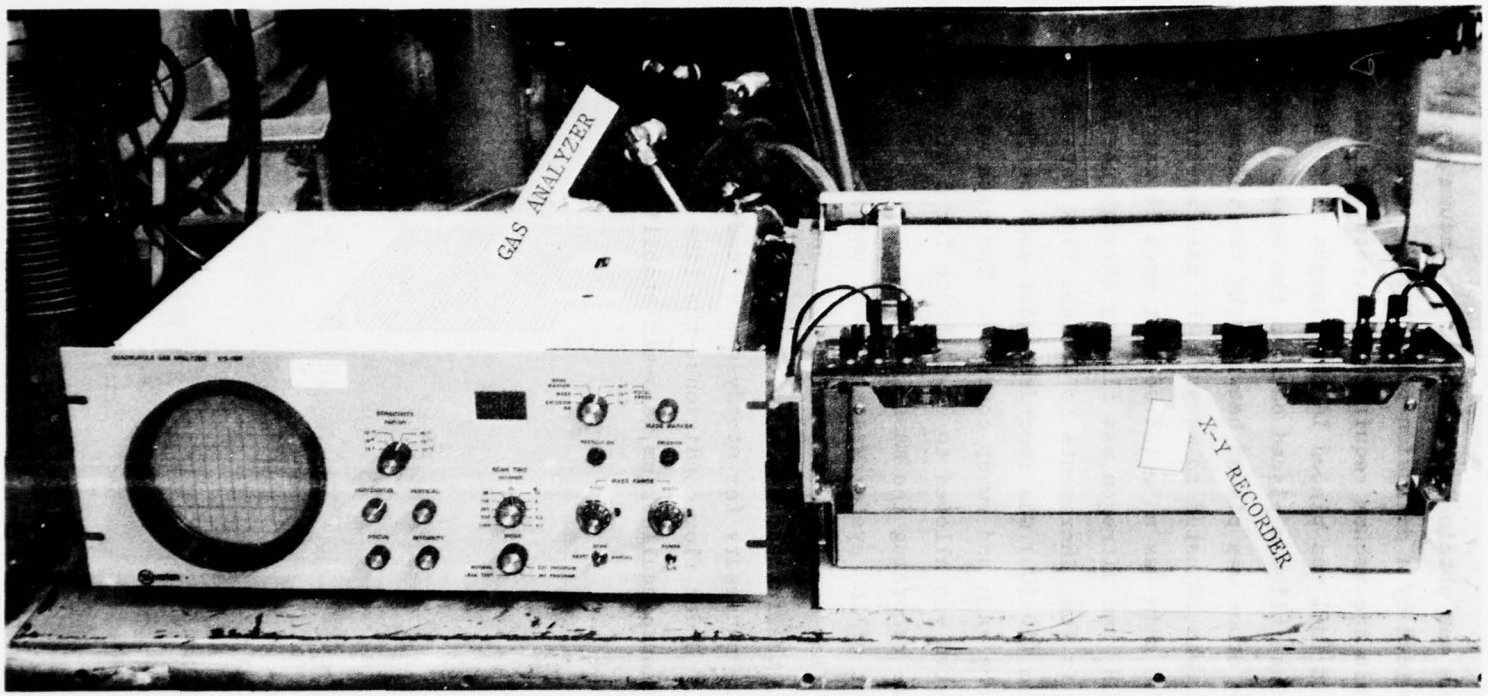

Fig. B-19. Gas analyzer control unit and X-Y recorder.

The first-stage vacuum was obtained from the house vacuum system, the second-stage was provided by a diffusion pump system. The two-stage system was necessary to obtain the vacuum required to operate the gas analyzer.

A perforated gold foil was placed in the flanges (Fig. B-20) to create a calibrated leak. One foil was placed between the exhaust gas and the first stage of pumping, the other was placed between the two vacuum systems.

The gas analyzer was adjusted to measure the mass spectrum from mass number 10 to 100. The gain was adjusted so that mass 16 was full-scale when a known quantity of CH_4 and nitrogen was flowing through the furnace. The CH_4 was then reduced in four increments to a final flow rate of zero. A complete mass spectrum (10-100) was recorded under each of the above conditions without readjusting the gain and sweep controls. The data were recorded on an X-Y plotter. The system was allowed to equilibrate for 5 min after each flow rate adjustment before recording the mass spectrum.

The time constant of the system was measured, and it was determined that 5 min was enough for flow equilibration. The time constant was measured by injecting helium into the furnace under no flow conditions (while at vacuum) and measuring the time necessary for steady-state reading on the gas analyzer. This time was <1 min. Under flow conditions, the time constant should be faster; however, to ensure equilubrium, a 5-min stabilization period was specified.

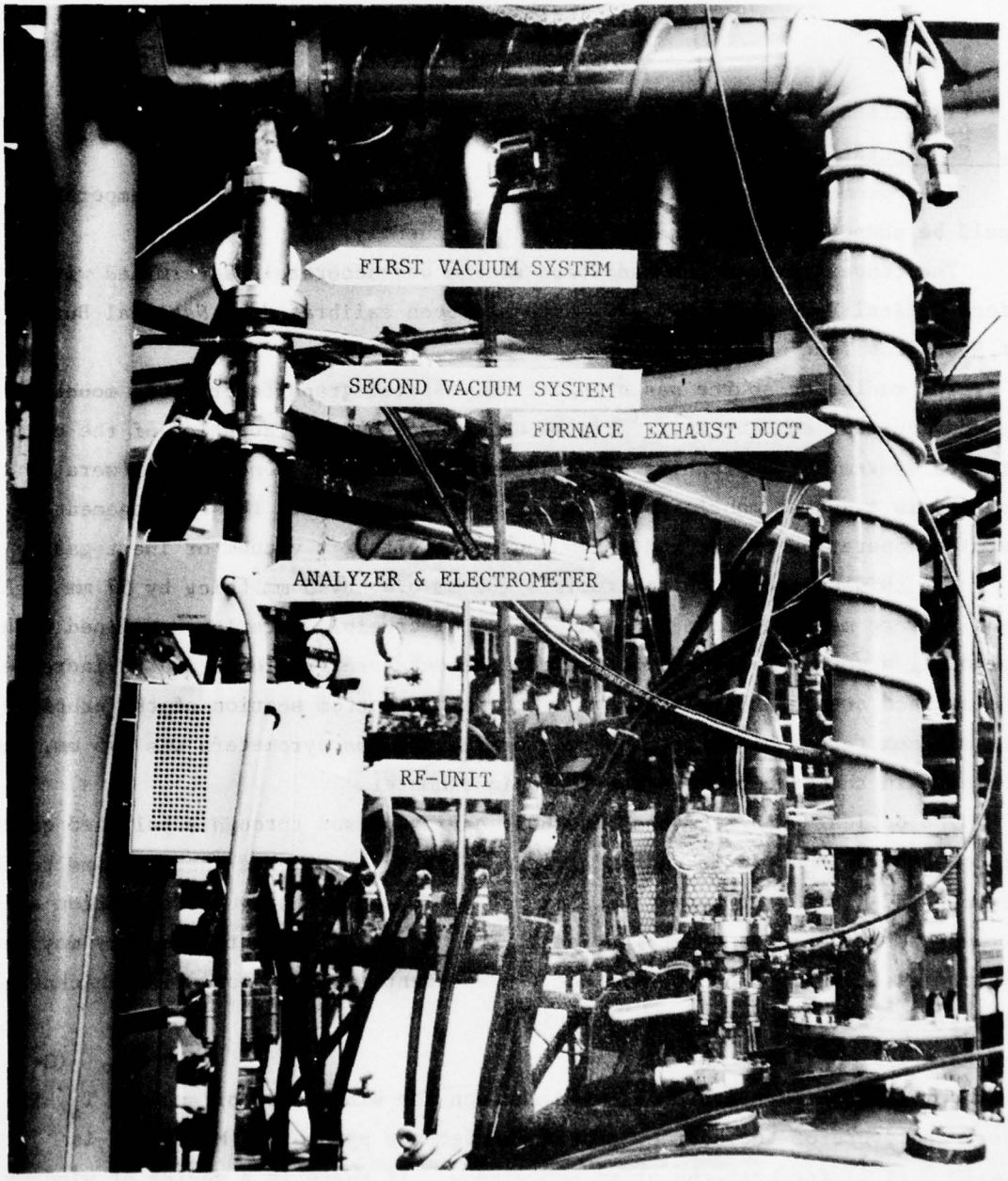

Fig. B-20. Gas analysis technique for furnace exhaust gas.

APPENDIX C

INSTRUMENT CALIBRATION

I. PYROMETERS

A. Configuration

The pyrometers were calibrated with a radiation source whose temperature could be changed readily and measured independently.

The temperature of the radiation source was accurately determined with a Micro Optical Pyrometer, M-5399, which had been calibrated by National Bureau of Standards Test No. 182836.

The radiation source was a inductively heated graphite crucible mounted in an eddy current concentrator. Figure C-1 shows the configuration of the calibration system. The graphite crucible and eddy current concentrator were mounted in a quartz mantle connected to a vacuum system. This arrangement permited operation of the radiation source in either a vacuum or inert gas.

The thin-walled graphite crucible (38 mm o.d. by 5 mm thick by 57 mm high) and lid were machined from ATJ graphite. The crucible interior was lined with Carbocel, a porous graphite, to minimize temperature gradients and to increase the surface area of the blackbody cavity in the bottom section of the crucible. The maximum target diameter required for the various pyrometers was 9.5 mm, and the hole in the crucible lid was 10 mm in diameter.

The optical path from the blackbody cavity passed through a polished quartz window and was then reflected at a right angle by a polished quartz prism to the pyrometers. The light beam intensity was decreased slightly by reflection as it passed through each quartz surface. The decrease in light intensity may be treated as an absorption coefficient (independent of window or prism thickness), and it was calculated from the relation

$$A = 1/T_o - 1/T_s \quad (K^{-1}), \qquad \text{(C-1)}$$

where T_o is the temperature observed through the window (or prism) and T_s, is the temperature of the source. For the reference pyrometer, M-5399, A is $2.6.10^{-6}$ (K^{-1}) for both the prism and window. If there is a series of windows or prisms in the optical path, i.e., n windows, the total absorption coefficient, A_t, is equal to nA. A will vary with the type of pyrometer, so the absorption coefficient was determined for each pyrometer calibrated and these are given in Table C-I. Further, quartz windows for the LASL deposition furnace were fabricated from the same quartz stock used for the calibration.

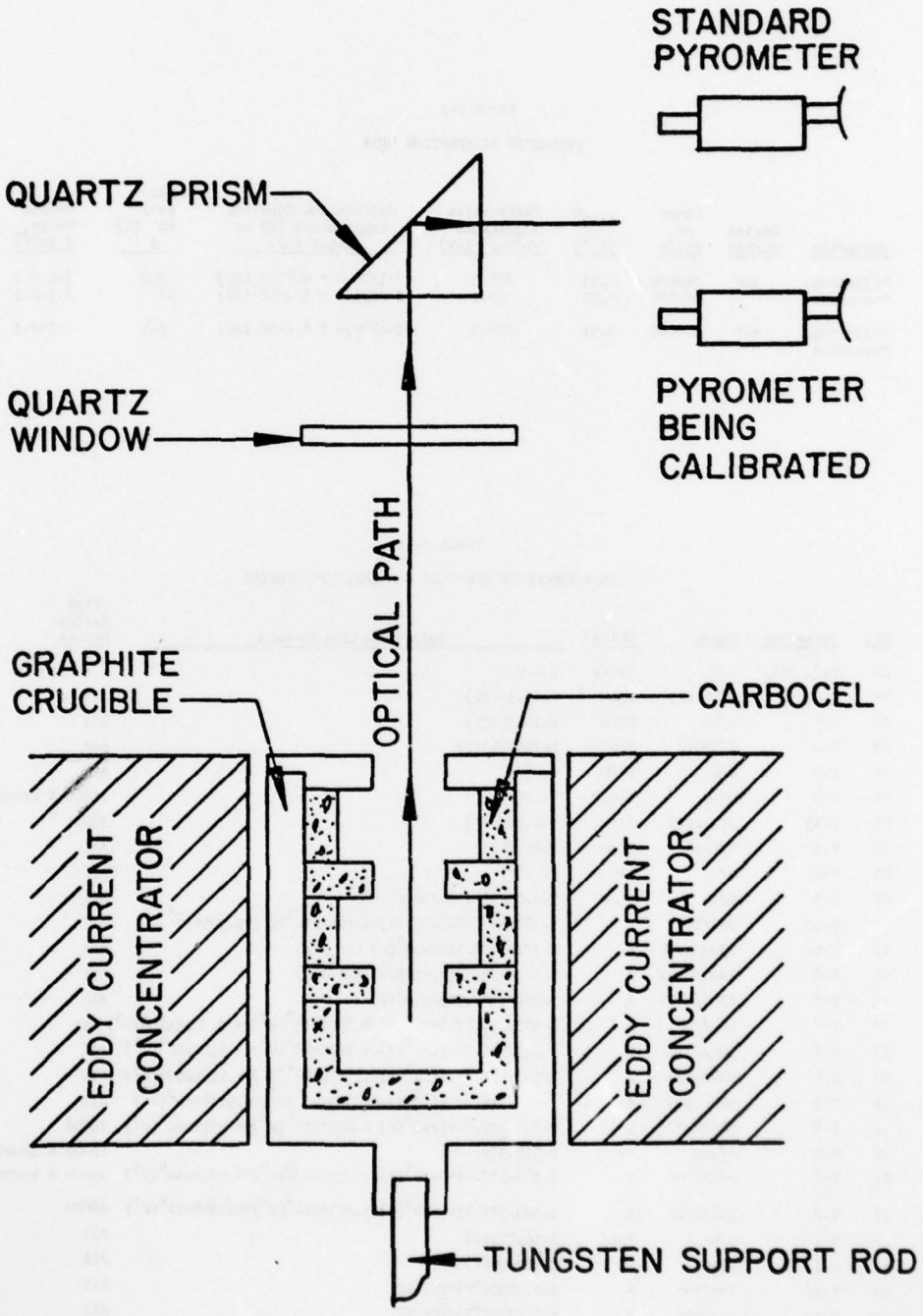

Fig. C-1. Configuration of calibration setup.

TABLE C-I

PYROMETER CALIBRATION DATA

Pyrometer	Serial Number	Range or Scale	$A \cdot 10^6$ (K^{-1})	Calibration Adjustment Voltage (mV)	Calibration Equation Temperature (K) vs Output (mV)	Std. Dev. $\pm\sigma$ (K) T	Calib. Range (10^{-3})
Milletron, Two-color	322	2000°C	0.91	100.5	T=1278.2 + 9.7189 (mV)	3.9	1.6-2.1
		3000°C	0.91	0.7	T=2295.0 + 12.942 (mV)	14	2.3-2.5
Milletron, Two-color	463	3000°C	0.51	232.0	T=1284.6 + 4.2232 (mV)	3.5	1.5-2.1

TABLE C-II

DATA REDUCTION FORMULAS FOR LASL TEST SERIES

Ch.	Parameter	Range	Units	Data Reduction Formula	Test Series Number
00	Ref. Cal.	---	Volts	Y = V	
01	F-8	0/46.175	SLPM	Y=15.399(V)	All
02	F-7	0/46	SLPM	Y=15.359(V)	All
03	F-6	0/340.6	SLPM	Y=115.655(V)	All
04	F-4	0/6	SLPM	Y=2(V)	All
05	F-5	0/30	¢ S/C	Y=10(V)	16000 & 17000
05	F-13	0/13.529	SLPM	Y=4.5098(V)	1800
06	F-1	0/1416	SLPM	Y=472(V)	All
07	F-2	0/9	GPM	Y=-3(V)	All
08	F-3	0/10	SLPM	Y=3.0329(V)+0.72897	All
09	T-10	500/1500	K	$Y=303.5+4.5868\times10^{2}(V)-4.7943\times10^{1}(V^2)+9.3952(V^3)$	All
10	T-6	1300/2500	K	$Y=1278.2+9.7198\times10^{3}(V)$ (Lo Rng)	All
10	T-6	1300/2500	K	$Y=12.942\times10^{3}(V)+2295.0$ (Hi Rng)	All
11	T-7	1300/2500	K	$Y=1284.6+4.2232\times10^{3}(V)$	All
12	T-2	500/2000	K	$Y=303.5+6.2276\times10^{3}(V)-8.8378\times10^{3}(V^2)+2.3515\times10^{4}(V^3)$	All
13	T-3	500/2000	K	$Y=303.5+6.2807\times10^{3}(V)-8.9892\times10^{3}(V^2)+2.4121\times10^{4}(V^3)$	All
14	T-4	500/2500	K	$Y=303.5+7.5515\times10^{3}(V)-1.2926\times10^{4}(V^2)+4.1594\times10^{4}(V^3)$	All
15	T-5	500/1500	K	$Y=303.5+4.637\times10^{3}(V)-4.8368\times10^{3}(V^2)+9.5204\times10^{3}(V^3)$	All
16	T-8	500/2500	K	$Y=303.5+7.4927\times10^{3}(V)-1.2793\times10^{4}(V^2)+4.0955\times10^{4}(V^3)$	16000
16	W-2	0/150	KW	Y=512.3(V)	17000 & 18000
17	T-9	500/2500	K	$Y=303.5+7.4748\times10^{3}(V)-1.2732\times10^{4}(V^2)+4.0662\times10^{4}(V^3)$	16000 & 17000
17	T-8	500/2500	K	$Y=303.5+7.4748\times10^{3}(V)-1.2732\times10^{4}(V^2)+4.0662\times10^{4}(V^3)$	18000
18	P-1	0/20	PSIA	Y=66.773(V)	All
19	W-1	0/150	KW	Y=500.00(V)	All
20	T-1	100/500	K	$Y=0.5556(^{\circ}F)+255.22$	All
21	T-11	273/310	K	$Y=0.5556(^{\circ}F)+255.22$	All
22-31	T12-T21	273/373	K	$Y=0.5556(^{\circ}F)+255.22$	All

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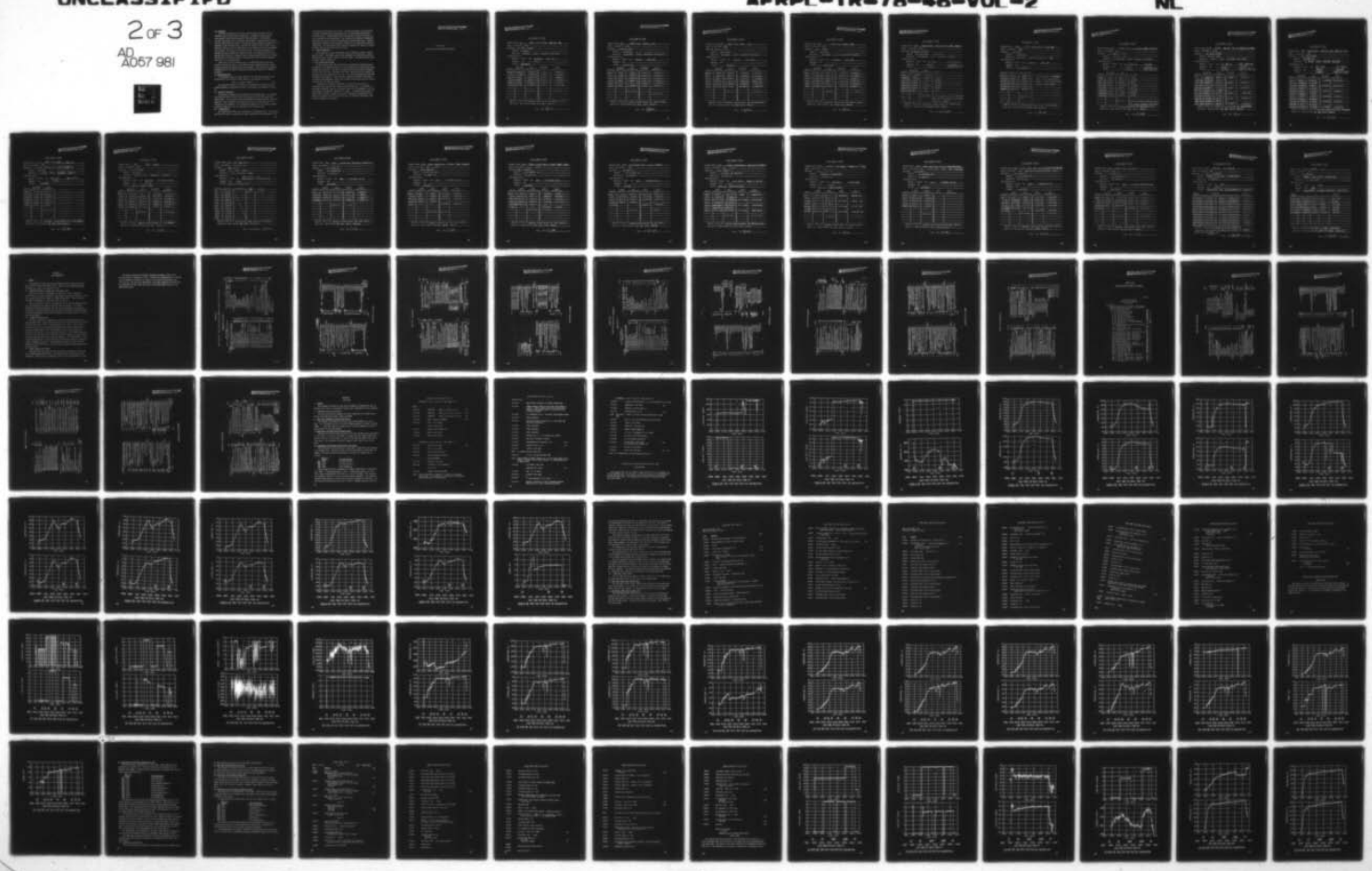

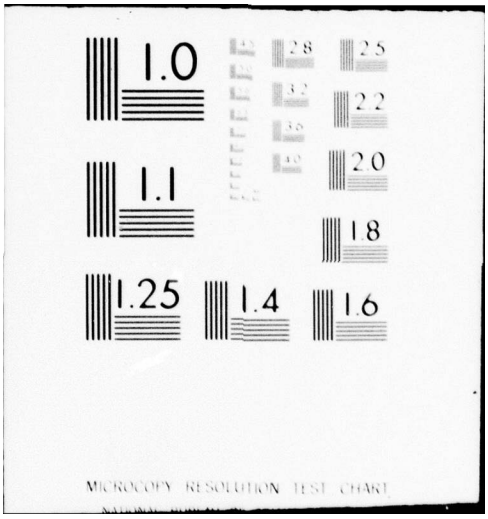
1.0
1.1
1.25
1.4
1.6
2.8
3.2
3.6
4.0
2.5
2.2
2.0
1.8
MICROCOPY RESOLUTION TEST CHART

B. Procedure

Specified preoperation, operation, and focusing procedures for each pyrometer were followed before calibration. Pyrometer voltage outputs were measured with a Hewlett-Packard 2490A multimeter. The blackbody crucible was brought up to temperature and allowed to equilibrate for 30 min, and the calibration was started. The voltage output and meter readings for the pyrometer being calibrated were recorded; the prism was then rotated to the reference pyrometer and the temperature of the radiation source was determined and recorded. The prism was then rotated back to the first pyrometer, and voltage and meter readings were again recorded. The temperature was raised sequentially (20-40 K), allowing 10 min for thermal equilization before repeating the above calibration steps, to approximately 2300 K and then sequenced back down to the starting point.

About every third calibration point, an additional quartz window was placed in the optical path to the pyrometer being calibrated and the change in meter reading and voltage output was recorded. This information permitted determination of the window's absorption coefficient relative to a specific pyrometer.

C. Calibration Data

The pyrometers data, mV output versus T, for one quartz window in the optical path, were fitted by least squares to an equation of the form

$$T(K) = A + B(mV) + C(mV)^2 + \ldots \qquad (C\text{-}2)$$

The standard deviation, σ_T, was used to determine what degree of polynominal adequately represented the data. The results are presented in Table C-I.

II. PRESSURE TRANSDUCER

The pressure transducer was calibrated by the manufacturer. The calibration data were used for the end-to-end calibration of the data acquisition system (DAS). The calibration was accomplished by disconnecting the electrical connector at the transducer and substituting a short circuit and then a dc voltage for the transducer. The calibration equation for the pressure channel is given in Table C-II for the nitrogen flow and coating tests.

III. THERMOCOUPLES

The thermocouples (TCs) were calibrated by the manufacturers. The calibration data were used to calibrate the signal conditioners and subsequently, for

the end-to-end calibration of the DAS. The DAS calibration for the W-5% Re vs W-26% Re channels was accomplished by the voltage substitution method described above. Voltage substitution was used for the type-T TCs also, but only as an end-to-end wiring check. The calibration consisted of immersing each Type-T TC in an ice bath and getting the resultant DAS output. Ambient-temperature readings of the units were compared for an additional calibration check point. The calibration (data reduction) equation for each TC is listed in Table C-II for the three series of tests.

IV. POWER TRANSDUCER

The voltage and current transformers were calibrated at LASL to verify their accuracy. The signal conditioning equipment was calibrated at LASL by varying the input voltage, current, and phase angle. The data reduction equations are shown in Table C-II.

V. DATA ACQUISITION SYSTEM

An end-to-end pretest DAS calibration was conducted as described above. In addition, the calibrate switches on the pyrometers were activated and these channels were verified. The above calibration data were recorded on magnetic tape for the first tests (nitrogen flow rates), and the data were reduced and verified before proceeding with the test. The calibration data were also hand-recorded on data sheets. A post-test DAS calibration similar to the pretest calibration was performed. Table C-III contains the pre and post-test calibration data for the LASL test series.

In addition to the above calibration, a pseudo pre- and post-test calibration was conducted just before and after each test. It consisted of recording on magnetic tape the calibration point for the pyrometers, zeros for some channels and ambient conditions for others. At least 10 data points for channel were recorded. Because of the need to maintain cooling water flow-rates on the furnace for ∿24 h after the test, no post-test calibration data on these channels were obtained.

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TABLE C-III

DATA ACQUISITION SYSTEM CALIBRATION

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CALIBRATION

FUNCTION F-1 MASS FLOW RATE PROCESS N_2

DAS CHANNEL 06

RANGE 0-1416 SLPM

INPUT 0-5VDC

OUTPUT 0-3VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. HASTINGS AHL-506

SERIAL NO. 361

TYPE THERMAL

INPUT	DIVIDER OUTPUT	DAS RECORDED	DIVIDER OUTPUT	DAS RECORDED
SHORT	.0000 V	.0002 V	0.000 V	0.000 V
1.0 V	1.6000	.6000 V	1.000 V	0.6002 V
2.0 V	1.200	1.2000 V	2.000 V	1.2001 V
3.0 V	1.800	1.8001 V	3.000 V	1.7999 V
4.0 V	2.400	2.4001 V	4.000 V	2.4007 V
5.0 V	3.000	2.9999 V	5.000 V	3.0001 V
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION F2 MASS FLOW RATE, MTS

DAS CHANNEL 07

RANGE 0-9 gms /MIN

INPUT 0-5VDC

OUTPUT 0-3VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. TYLAN GP-348

SERIAL NO.

TYPE THERMAL

INPUT	DIVIDER OUTPUT	DAS RECORDED	DIVIDER OUTPUT	DAS RECORDED
SHORT	.0000	.0003V	0.000 V	0.0001V
-1.0V	-.6000	-.6001 V	-0.600 V	-0.6003 V
-2.0V	-1.200	-1.2002 V	-1.200 V	-1.2001 V
-3.0V	-1.800	-1.8000 V	-1.800 V	-1.8002 V
-4.0V	-2.400	-2.4001 V	-2.400 V	-2.4003 V
-5.0V	-3.000	-2.9999 V	-3.000V	-3.0002 V
		8-19-76		
				11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER

OUTPUT MEASURED AT DAS INPUT TERMS

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CALIBRATION

FUNCTION F3 MASS FLOW RATE CH_4

DAS CHANNEL 08

RANGE 0-10 SLPM

INPUT 0-5 VDC

OUTPUT 0-3 VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. TYLAN GP-348

SERIAL NO.

TYPE THERMAL

INPUT	DIVIDER OUTPUT	DAS RECORDED	DIVIDER OUTPUT	DAS RECORDED
SHORT	.0000 V	.0001 V	0.0000 V	0.0001 V
1.0 V	.6000 V	.6001 V	0.6000 V	0.6001 V
2.0 V	1.200 V	1.1999 V	1.2000 V	1.2008 V
3.0 V	1.800 V	1.7999 V	1.8000 V	1.8007 V
4.0 V	2.400 V	2.3963 V	2.4000 V	2.4007 V
5.0 V	3.000 V	2.9969	3.0000 V	3.0009 V
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY GCM

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CALIBRATION

FUNCTION F4 MASS FLOW RATE, He

DAS CHANNEL 04

RANGE 0-6 SLPM

INPUT 0-5 VDC

OUTPUT 0-3 VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. TYLAN GP-348

SERIAL NO.

TYPE THERMAL

INPUT	DIVIDER OUTPUT	DAS RECORDED	DIVIDER OUTPUT	DAS RECORDED
SHORT	.0000 V	.0002 V	.0000 V	0.0001 V
1.0V	.6000 V	.6001 V	.6000 V	0.5993V
2.0V	1.200 V	1.2003 V	1.200 V	1.1981 V
3.0V	1.800 V	1.8001 V	1.800 V	1.7977 V
4.0V	2.400 V	2.4001 V	2.400 V	2.3970 V
5.0V	3.000 V	3.0000 V	3.000 V	2.9960 V
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION F-6 FLOW RATE; COOLING H_2O, BELL JACKETS

DAS CHANNEL 03

RANGE 0-340.6 SLPM

INPUT 0-1553 HZ 0-90 GPM

OUTPUT 0-3 VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. COX-AN-20 F.T.I PRIH02A

SERIAL NO. 29692 150883

TYPE TURBINE FREQ. TO DC CONVERTER & INDICATOR

INPUT	DIVIDER OUTPUT	DAS RECORDED	METER
SHORT	-.0025	-.0024	0
300 HZ	.5846	.5847	17.5%
600 HZ	1.1672	1.1672	34.8%
900 HZ	1.7460	1.7461	52.2%
1200 HZ	2.3226	2.3228	69.6%
1500 HZ	2.8989	2.8991	87%
1553 HZ	3.0001	3.0002	90%
		11-17-76	THIS TURBINE PICKUP REPLACED
			F.T.I. P/N FT-20M90-LB FOR TESTS
			1700- & 1800—

INPUT APPLIED SIG. GEN. SUBSTITUTION AT TRANSDUCER

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION F5 FLOW RATE; RATIO, MTS/He

DAS CHANNEL 05

RANGE 0-30%

INPUT 0-5 VDC

OUTPUT 0-3 VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. TYLAN GP-348

SERIAL NO.

TYPE CALCULATED

INPUT	OUTPUT	RECORDED	
SHORT	.0000	.0004 V	MEASUREMENT REPLACED
1.000 V	.6000 V	.6001 V	BY F-13 FOR TEST
2.000 V	1.200 V	1.2001 V	1800-. NOT CALIBRATED
3.000 V	1.800 V	1.8001 V	AFTER LAST TEST.
4.000 V	2.400 V	2.4002 V	
5.000 V	3.000 V	3.0001 V	
		8-19-76	

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER

OUTPUT MEASURED AT DAS INPUT TERMS.

CAL. BY JCM

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CALIBRATION

FUNCTION F6 FLOW RATE; COOLING H_2O, BELL JACKETS

DAS CHANNEL 03

RANGE 0-340.6 SLPM

INPUT 0-2012 HZ

OUTPUT 0-3.0 VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. FT 20M90-LB PRI-102A

SERIAL NO. 20039 150883

TYPE TURBINE FREQ. TO DC CONVERTER AND INDICATOR

INPUT	DIVIDER OUTPUT	DAS RECORDED	METER
0 HZ	.005	.005 V	0%
300 HZ	.4444	.4445 V	14.5%
600 HZ	.8865	.8868 V	29%
900 HZ	1.326	1.3270V	44.5%
1200 HZ	1.7679	1.7681	60%
1500 HZ	2.2032	2.2039	74.5%
1800 HZ	2.6399	2.6398	89%
2012 HZ	2.9449	2.9453	100%
		8-19-76	TURBINE PICKUP FAILED DURING TEST 16000, WAS REPLACED WITH COX P/N AN-20 S/N 24642

INPUT APPLIED SIG. GEN. SUBSTITUTION AT TRANSDUCER

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION F-7 FLOW RATE; COOLING H_2O, CENTER BODY

DAS CHANNEL 02

RANGE 0-47.3 SLPM

INPUT 0-1200 HZ

OUTPUT 0-3 V FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. Cox AN-10 FTI. PRC101

SERIAL NO. 24811 86757

TYPE TURBINE FREQ. TO DC CONVERTER

INPUT	DIVIDER OUTPUT	DAS RECORDED	DIVIDER OUTPUT	DAS RECORDED
0	000.0V	.006 V	0.000V	.0068 V
120 HZ	.3092V	.3092 V		
240 HZ	.6095V	.6096 V	.6103	.6104 V
360 HZ	.9089V	.9090 V		
480 HZ	1.2088V	1.2089 V	1.2105	1.2105 V
600 HZ	1.5082 V	1.5084 V		
720 HZ	1.8047 V	1.8048 V	1.8067	1.8068 V
840 HZ	2.1042 V	2.1044 V		
960 HZ	2.4005 V	2.4007 V	2.4031	2.4031 V
1080 HZ	2.6984 V	2.6985 V		
1200 HZ	2.9950 V	2.9950 V	2.9985	2.9986 V

INPUT APPLIED 8-19-76 SIG. GEN. SUBSTITUTION AT TRANSDUCER 11-17-76

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION F-8 FLOW RATE: COOLING H_2O, HEATING COIL

DAS CHANNEL 01

RANGE 0 - 47.3 SLPM

INPUT 0-1200 HZ

OUTPUT 0-3VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO.	LOX AN-10	FTI PRC-101
SERIAL NO.	24812	860758
TYPE	TURBINE	FREQ TO DC. CONV.

INPUT	DIVIDER OUTPUT	DAS RECORDED	DIVIDER OUTPUT	DAS RECORDED
SHORT	.00512 V	0.0051V	0.0055V	0.0055 V
120 HZ	.30885V	0.3089V		
240 HZ	.60908V	0.6091 V	0.6102 V	0.6104 V
360HZ	.91051 V	0.9106 V		
480HZ	1.2092 V	1.2094 V	1.2127 V	1.2128 V
600HZ	1.5087 V	1.5090 V		
720HZ	1.8074 V	1.8077 V	1.8128 V	1.8130 V
840HZ	2.1063 V	2.1066 V		
960HZ	2.4035V	2.4038V	2.4079 V	2.4080 V
1080 HZ	2.7017 V	2.7019 V		
1200 HZ	2.9983V	2.9968V	3.0068V	3.0070V

INPUT APPLIED 8-19-76 SIG. GEN. SUBSTITUTION 11-17-76 AT TRANSDUCER

OUTPUT MEASURED AT DAS INPUT TERMS

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CALIBRATION

FUNCTION F-13 MASS FLOW RATE ; AUX CH_4

DAS CHANNEL 05

RANGE 0-20K SLPM N_2 = 0-13.5294 SLPM CH_4

INPUT 0-5 VDC

OUTPUT 0-3VDC FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. HASTINGS ALL 50 KGX

SERIAL NO. 2172

TYPE THERMAL

INPUT	DIVIDER OUTPUT	DAS RECORDED	
SHORT	0.000V	0.0001 V	
1.000 V	0.600V	0.6003 V	
2.000 V	1.200 V	1.2007 V	
3.000 V	1.800 V	1.8011 V	
4.000 V	2.400 V	2.4009 V	
5.000 V	3.000 V	3.009 V	
		11-17-76	

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER.

OUTPUT MEASURED AT DAS INPUT TERMS.

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CALIBRATION

FUNCTION P-1 PRESS. STATIC

DAS CHANNEL 18

RANGE 0-20 PSIA

INPUT 0-5 V

OUTPUT 0-300mV FROM VOLTAGE DIVIDER

TRANSDUCER

MFGR.— PART NO. STD CONT 212-25-010-13

SERIAL NO. 30669

TYPE BONDED STRAIN GAGE

INPUT	XDUCER OUTPUT	DAS RECORDED	XDUCER OUTPUT	DAS RECORDED
SHORT	.0000 V	0.9 mV	.00000 V	0.01 mV
1.0 V	.06000 V	060.00 mV	.05997 V	59.99 mV
2.0 V	.12000 V	120.00 mV	.11997 V	119.98 mV
3.0 V	.18000 V	179.99 mV	.17993 V	179.99 mV
4.0 V	.24000 V	240.03 mV	.23995 V	239.95 mV
5.0 V	.30000 V	299.99 mV	.29986 V	299.87 mV
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT TRANSDUCER

OUTPUT MEASURED AT DAS INPUT TERMS.

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CALIBRATION

FUNCTION T1 & T11 THRU T21

DAS CHANNEL 20 THRU 31

RANGE 100 - 373°K

INPUT ICE & WATER BATH

OUTPUT DAS RECORDED VALUE

TRANSDUCER

MFGR.— PART NO. ARI* T-91R-32 FT9C - THERMO ELECTRIC* T18U-304-0-12-0L

SERIAL NO. NONE

TYPE TYPE "T" THERMOCOUPLE

INPUT	OUTPUT	RECORDED	INPUT	OUTPUT
T-1	31.6°F		T-21	31.2°F
T-11	31.2°F			
T-12	31.2°F			
T-13	31.1°F			
T-14	30.5°F			
T-15	30.8°F			
T-16	31.2°F			
T-17	31.2°F			
T-18	31.3°F			
T-19	31.5°F			
T-20	31.6°F			

INPUT APPLIED THERMOCOUPLES WERE IMMERSED IN ICE BATH

OUTPUT MEASURED BY DAS PRINTOUT

CAL. BY QCM 8-23-76

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CALIBRATION

FUNCTION T-2 TEMP, WITHIN WALL OF INLET TUBE (BOTTOM)

DAS CHANNEL 12 K2

RANGE 500 - 2000° K

INPUT 0 - 29.705 mV

OUTPUT 0 - 300 mV

TRANSDUCER

MFGR.— PART NO. A.R.I. TS0386-12-30

SERIAL NO.

TYPE W/WRe

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.0003 V	.0003 V	-.00015 V	-.00015 V
10.00 mV	10.11 V	.1011 V	+.10029 V	+.10029 V
20.00 mV	.2009 V	.2009 V	+.20071 V	+.20071 V
29.705 mV	.2990 V	.2990 V	+.29811 V	+.29811 V
		8-19-76	11-17-76	

INPUT APPLIED VOLTAGE SUBSTITUTION AT T/C INPUT

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY C. M

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CALIBRATION

FUNCTION T3 TEMP: WITHIN WALL of INLET TUBE (MIDDLE)

DAS CHANNEL 13 K2

RANGE 500-2000°K

INPUT 0-29.705 mV

OUTPUT 0-300 mV

TRANSDUCER

MFGR.— PART NO. ARI T50386-12-30

SERIAL NO.

TYPE W/WRe

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.00009 V	.0000 V	.00009 V	.00009 V
10.0 mV	.098 V	.0980 V	.10503 V	.10504 V
20.0 mV	.1993 V	.1994 V	.21014 V	.21014 V
29.705 mV	.2983 V	2983 V		
28.56 mV	—	—	.29956 V	.29957 V
		8-19-76	11-17-76	

INPUT APPLIED VOLTAGE SUBSTITUTION AT T/C INPUT

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY [signature]

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CALIBRATION

FUNCTION T 9 TEMP.; WITHIN WALL OF INLET TUBE (TOP)

DAS CHANNEL 19 K 2

RANGE 50C - 2589°K

INPUT 0 - 35.842 mV

OUTPUT 0 - 300 mV

TRANSDUCER

MFGR.— PART NO. ARI T50386-12-30

SERIAL NO. ____

TYPE W/WRe

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.59 mV	.0007 V	.00009V	.00006 V
10.0 mV	8107.2 mV	.0817 V	.08381V	.08382 V
20.0 mV	165.5 MV	.1654 V	.16760V	.16760 V
30.0 mV	299.3 mV	.2993 V	.25136V	.25136 V
35.842 mV	298.2 mV	.2981 V	—	
35.82 mV	—	—	.30008 V	.30008 V
		8-19-76	11-17-76	

INPUT APPLIED VOLTAGE SUBSTITUTION AT T/C INPUT

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY C M

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CALIBRATION

FUNCTION T5 TEMP; CARBON FELT OUTSIDE SURFACE

DAS CHANNEL 15

RANGE 0-1500° K

INPUT 0-21.919 mV

OUTPUT 0-300 mV

TRANSDUCER

MFGR.— PART NO. ARI T50386-9-33

SERIAL NO.

TYPE W/WRe

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.9 mV	.0005 V	.00009 V	.00013 V
10 mV	134.9 mV	.1348 V	.13597 V	.13597 V
20 mV	271.7 mV	.2716 V	.27206 V	.27206 V
21.919 mV	298.0 mV	.2980 V		
22.05 mV			.30000 V	.30000 V
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT T/C INPUT

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION T 6 TEMP: SUBSTRATE BACKSIDE SURFACE

DAS CHANNEL 10 - K2

RANGE 1300 - 2500° K

INPUT 14.15 mV @ 1915.7°K

OUTPUT 0 - 300 mV

TRANSDUCER

MFGR.— PART NO. MILLITRON THERM-O-SCOPE

SERIAL NO. 322

TYPE OPTICAL PYROMETER

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.0000	000.34 mV	.0000	000.03 mV
50 mV	50.00 mV	99.69 mV		
100 mV	100.00 mV	99.68 mV	100.00 mV	100.07 mV
150 mV	150.00 mV	199.59 mV		
200 mV	200.00 mV	199.63 mV	200.00 mV	200.07 mV
250 mV	250.00 mV	249.70 mV		
300 mV	300.00 mV	299.74 mV	300.00 mV	300.06 mV
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER

OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION T 7 TEMP: SUBSTRATE DEPOSITION SURFACE
DAS CHANNEL 11
RANGE 1300 - 2500°K
INPUT 232 mV @ 2264.4°K
OUTPUT 0-300 mV
TRANSDUCER
MFGR.— PART NO. MILLITRON THERMOSCOPE
SERIAL NO. 463
TYPE OPTICAL PYROMETER

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
START	.0000 mV	000.01 mV	000.00 mV	00.00 mV
50 mV	50.00 mV	49.51 mV		
100 mV	100.0 mV	99.63 mV	100.00 mV	99.99 mV
150 mV	150.0 mV	149.69 mV		
200 mV	200.0 mV	199.73 mV	200.00 mV	200.00 mV
250 mV	250.0 mV	249.69 mV		
300 mV	300.0 mV	299.77 mV	300.00 mV	300.09 mV
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT CONDITIONER
OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION T8 TEMP WITHIN WALL of EXIT TUBE (BOTTOM)
DAS CHANNEL 16 FOR TEST 1600- ONLY (FAILED)
RANGE 0-2500°K
INPUT 0-35.892 mV
OUTPUT 0-300 mV
TRANSDUCER
MFGR.— PART NO. AR1 T5038C-12-30
SERIAL NO.
TYPE W/WRe

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.0000 V	.0001 V		
10 mV	83.4 mV	.0833 V		
20 mV	167.02 mV	.1670 V		
30 mV	250.6 mV	.2506 V		
35.892 mV	299.5 mV	.2995 V		
	—	—		
		8-19-76		

INPUT APPLIED VOLTAGE SUBSTITUTION AT T/C INPUT,
OUTPUT MEASURED AT DAS INPUT TERMS

CAL. BY JCM

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CALIBRATION

FUNCTION T9 TEMP WITH WALL of EXIT TUBE (180° FROM T8)

DAS CHANNEL 17 INSTALLED IN T-8 POSITION FOR TEST 1800—

RANGE 0 - 2500°K

INPUT 0 - 35.842 mV

OUTPUT 0 - 300 mV

TRANSDUCER

MFGR.— PART NO. AIRI T50386-12-30

SERIAL NO.

TYPE W/WRe

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.0000	.0000 V	.00007 V	.00009 V
10 mV	83.8 mV	.0839 V	.08679 V	.08678 V
20 mV	167.5 mV	.1679 V	.17342 V	.17343 V
30 mV	251.2 mV	.2513 V	.26006 V	.26006 V
35.842 mV	300.1 mV	.3004 V	—	
34.61 mV	—	—	.30005 V	.30005 V
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT T/C INPUT.

OUTPUT MEASURED AT DAS INPUT TERMS.

CAL. BY QCM

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CALIBRATION

FUNCTION T10 TEMP GAS EXHAUST

DAS CHANNEL 09

RANGE 0-1500° K

INPUT 0-21.919 mV

OUTPUT 0-3.000 V

TRANSDUCER

MFGR.— PART NO. ARI T-99B-12DAE9N300

SERIAL NO. —

TYPE W/WRe

INPUT	AMP OUTPUT	DAS RECORDED	AMP OUTPUT	DAS RECORDED
SHORT	.0004	.0007 V	.0002	.00025 V
10 mV	1.3639	1.3681 V	1.3631	1.3635 V
20 mV	2.729	2.7265 V	2.7241	2.7243
21.919 mV	2.991	2.9919 V	—	—
22.030 mV	—	—	2.9995	2.9996 V
		8-19-76		11-17-76

INPUT APPLIED VOLTAGE SUBSTITUTION AT T/C INPUT

OUTPUT MEASURED AT DAS INPUT TERMS.

CAL. BY [illegible]

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CALIBRATION

FUNCTION W-1 POWER E·I COS Ø

DAS CHANNEL 19

RANGE K2 0-150 KW

INPUT FURNACE VOLTAGE /B & CURRENT /750

OUTPUT 0-300.0 mv

TRANSDUCER

MFGR.— PART NO. LASL

SERIAL NO. NONE

TYPE TRANSFORMER AND SIGNAL CONDITIONER - MULTIPLIER

SIMUL. INPUT	E-IN	I-IN	*PHASE ANGLE	OUTPUT	CALC K-W	DAS RECORDED
149.876 KW	399.4 V	375.3 A	0°	.30354 V	149.876 KW	303.54 mV
142.712 KW	399.4 V	375.76 A	+18°	.28955 V	142.968 KW	289.55 mV
142.538 KW	399.38 V	375.28 A	-18°	.28592 V	147.176 KW	285.92 mV
96.117 KW	320.13 V	300.22 A	0°	.19162 V	94.614 KW	191.62 mV
91.538	320.13 V	300.65 A	+18°	.18238 V	90.052 KW	182.38 mV
78.023	320.13 V	301.29 A	+36°	.15522 V	76.691 KW	155.22 mV
91.394 KW	320.13 V	300.18 A	-18°	.18252 V	90.121 KW	182.52 mV
77.798 KW	320.13 V	300.37 A	-36°	.15476 V	76.414 KW	154.76 mV
71.986 KW	320.13 V	224.85 A	0°	.14260 V	70.410 KW	142.60 mV
68.568 KW	320.13 V	225.21 A	+18°	.13480 V	66.559 KW	134.80 mV
68.429 KW	320.13 V	224.75 A	-18°	.13667 V	67.482 KW	136.67 mV

INPUT APPLIED AT INPUT OF SIGNAL CONDITIONER

OUTPUT MEASURED AT OUTPUT OF SIGNAL CONDITIONER

* PHASE ANGLE = 'I' LEADING IS +
'I' LAGGING IS -

CAL. BY JCM 10-5-76

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CALIBRATION

FUNCTION W-2 POWER E·I

DAS CHANNEL 16

RANGE 0 - 150 KW

INPUT FURNACE VOLTAGE/8 & CURRENT/750

OUTPUT 0 - 300.0 mV

TRANSDUCER

MFGR.— PART NO. LASL

SERIAL NO. NONE

TYPE TRANSFORMER & SIGNAL CONDITIONER - MULTIPLIER

SIMUL INPUT	E-IN	I-IN	OUTPUT	CALC KW	DAS RECORDED
149.939 KW	400.32 V	374.55 I	.2928 V	150.00	292.8 mV
95.287 KW	319.84 V	306.05 I	.1879 V	96.26	187.9 mV
54.072 KW	249.16 V	225.15 I	.1055 V	54.047	105.5 mV
24.156 KW	160.88 V	150.15 I	.0471 V	24.129	47.1 mV
36.21 KW	160.88 V	225.075 I	.0705 V	36.117	70.5 mV
36.096 KW	240.4 V	150.15 I	.0704 V	36.065	70.4 mV

INPUT APPLIED AT INPUT OF SIGNAL CONDITIONER

OUTPUT MEASURED AT OUTPUT OF SIGNAL CONDITIONER

CAL. BY JCM 10-5-76

APPENDIX D
TEST PROCEDURES

I. GENERAL

This appendix contains the test procedures (Tables D-I-D-III) for the three series of tests performed at LASL. These include transient heating, nitrogen flow, power, and deposition tests.

II. TRANSIENT HEATING, NITROGEN FLOW, AND POWER TESTS

These tests started on September 20, (day 264), 1976 at 1036 after several hours of system configuration verification and pretest check-out. The test series identification number is 16000 through 16005. These tests were concluded on September 21, (day 265), 1976, at 2018 h.

The test procedure Table D-I was modified as the test proceeded and data were being analyzed and compared with the test objectives. As a result, paragraph 2.8.18 was modified in that the test was allowed to proceed with a lower furnace vacuum and temperature than that required by the test procedure. It was also determined that it was not necessary to perform items 3.3-3.3.10 of the test procedure.

III. POWER AND COATING TESTS

The test identification numbers for the power and deposition tests are 17000 through 17010. These tests started at 1545 on day 287 (October 18, 1976) and were concluded at 1820 h on day 288 (October 14, 1976). As the tests proceeded, the data were analyzed and compared with the test objectives and the test procedure Table D-II, was modified to fulfill these objectives. Because of the failure of a cooling water flow meter, the procedure described in item 2.6 was postponed until after the furnace cool-down, at which time the flow meter was repaired and the test was conducted. The requirements of item 2.8 were performed over a 2-day period; so the DAS was not used to record the furnace outgassing procedure. The procedures outlined in item 2.8.16 were modified to allow the tests to proceed at lower vacuums and temperatures than those specified.

IV. SECOND COATING TEST SERIES

The identification numbers of this second group of deposition tests are in the 18000 through 18009 series. The tests started at 1012 h on day 313 (November 8, 1976) and were concluded at 1200 h on day 314 (November 9, 1976).

The pretest checkout and furnace outgassing procedure, Table D-III, started on day 310 (November 5, 1976). Because the outgassing was to continue over the weekend, the DAS did not monitor this activity continuously (see 2.8.1-2.8.4). the water flow measurement tests were conducted on day 309 and are identified as test 17000; they were not performed again as part of the 18000 series tests.

TABLE D-I

TEST PROCEDURE - TRANSIENT HEATING, N_2 FLOW, AND POWER TEST PROCEDURES

OPERATOR'S PROCEDURE

LASL COATING FURNACE

TRANSIENT HEATING, N_2 FLOW, AND POWER TEST PROCEDURES

September 9, 1976

1.0 Assembly

1.1 Fixtures assembled as per 26 Y 199115. Check following items for proper location, alignment, and assure that correct transducers are in place.

1.1.1	T-1	Gas-Furnace Inlet		
.2	T-2	Bottom Wall Inlet Tube		
.3	T-3	Middle Wall Inlet Tube		
.4	T-4	Top Wall Inlet Tube		
.5	T-5	Outside Surf. Carbon Felt		
.6	T-6	Back Surf. Substrate		
.7	T-7	Deposition Surf. Substrate		
.8	T-8	Bottom Wall Exit Tube BAD THERMOCOUPLE	±	±
.9	T-9	180° from T-8		
.10	T-10	Gas Exhaust (Thermocouple at full insertion minus ½ inch)		
.11	T-11	Cooling Water Supply		
.12	T-12	Cooling Water Return - Top & Mid Bell Jar Jackets		
.13	T-13	Cooling Water Return - Heating Coil Support		
.14	T-14	Cooling Water Return - Center Body		
.15	T-15	Cooling Water Return - Base Bell Jar		
.16	T-16	Cooling Water Return - Precooler Coil		
.17	T-17	Cooling Water Return - Heating Coil		
.18	T-18	Cooling Water Return - Top Canopy		
.19	T-19	Room Temperature		
.20	T-20	Cooling Water Return - Capacitor		
.21	T-21	Cooling Water Return - Capacitor Bus Bar		
.22	P-1	Lower Plenum Pressure		
.23	F-1	Mass Flow - Process N_2		
.24	F-2	Mass Flow Rate - MIS		
.25	F-3	Mass Flow Rate - CH_4		
.26	F-4	Mass Flow Rate - He		
.27	F-6	Flow Rate - Cooling Water - Top & Mid Bell Jar Jacket		
.28	F-7	Flow Rate - Cooling Water - Center Body		
.29	F-8	Flow Rate - Cooling Water - Heating Coil		
.30	F-9	Flow Rate - Cooling Water - Heating Coil Support		

1.1.31	F-10	Flow Rate - Cooling Water - Base Bell Jar		
.32	F-11	Flow Rate - Cooling Water - Precooler Coil		
.33	F-12	Flow Rate - Cooling Water - Top Canopy		
.34	W-1	Power EI Cos ϕ		
.35	MS-1	Mass Spectrometer	±	±

1.2 Cooling water connections to:

1.2.1	Top & Mid Bell Jar Jackets	—	
.2	Heating Coil Support	—	
.3	Center Body	—	
.4	Base Bell Jar	—	
.5	Precooler Coil	—	
.6	Heating Coil	—	
.7	Top Canopy	—	
.8	Capacitor Bus Bar	—	
.9	Capacitor Bus Bar	—	
.10	Power Supply	—	
.11	Mass Spectrometer Mount	±	±

1.3 Process gas connections for:

1.3.1	Process N_2	—	
.2	CH_4 (to Tylan Panel)	±	—
.3	He (to Tylan Panel)	±	—
.4	House N_2 (to Tylan Panel)	±	—
.5	MIS	±	—
.6	Air (to Tylan Panel for pneumatic valves)	±	—

1.4	Initial MIS System Purge	—	
1.5	Purge gas connection to T-7	—	
1.6	Furnace Power connections	—	
1.7	Capacitor connections made and recorded	—	
1.8	Turn on all instrumentation and power supplies. Place pyrometers in "CAL" mode (DAS procedure 1.1)		—
1.9	Set Test ID number to 16000. (See DAS procedures 2.1 and 1.2)		—
1.10	After 1 hr perform 10 complete data scans. Print out 1st and last scans. (See DAS procedure 1.4) Insert 1 file gap (DAS procedure 2.2.1) on magnetic tape		—

2.0 Pre-Heatup Check Out:

2.1	Process and House N_2 Supply	—	
2.2	CH_4 Supply	±	—

* Not used in these tests.

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TABLE D-I (continued)

2.3 MTS Supply ± —
2.4 He Supply ± —
2.5 Air Supply ± —
2.6 Water flow rate measurement:
2.6.1 Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-12, F-9, F-6)
.2 Open WR-1
.3 Close WS-2
.4 Open WS-3
.5 Close WS-4
.6 Close WS-5
.7 Open WS-6
.8 Open WS-7
.9 Close WS-8
.10 Close WS-10
.11 Close WS-11
.12 Open WS-9
.13 Open WS-12
.14 Open WS-1
.15 Initiate DAS for continuous scan of above channels and take a minimum of 10 complete scans prior to proceeding to next step. (DAS procedure 1.4) Print out the first two scans of data (DAS procedure)
.16 Record DAS time 10:26:00 Day 264
.17 Discontinue DAS scan (DAS procedure 1.4.1) Insert 1 file gap on magnetic tape (DAS procedure 2.2.1)
.18 Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-11, F-10, F-6)
.19 Close WS-3
.20 Open WS-2
.21 Close WS-6
.22 Open WS-5
.23 Close WS-9
.24 Open WS-8
.25 Close WS-12
.26 Open WS-11
.27 Repeat 2.6.15
.28 Record DAS time 10:39:00 Day 264

+ Not used in these tests.

2.6.29 Repeat 2.6.17
.30 Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-8, F-7, F-6)
.31 Close WS-5
.32 Open WS-6
.33 Open WS-4
.34 Close WS-11
.35 Open WS-10
.36 Open WS-12
.37 Repeat 2.6.15
.38 Record DAS time 10:42:00 Day 264
.39 Repeat 2.6.17
2.7 LASL (SM-29) House Services set up and checked off as follows:
2.7.1 N_2(Process) trailer full C
.2 CNC-2 service shut off and Group notified. ± ±
.3 Trailer connected, manifold purged, delivery pressure set at 75 psig. C
.4 Process N_2 pressure gage at flow meter checked at 4/ psig. C
.5 House N_2 (purge) manifold connected and regulator delivery pressure set at 75 psig. C
.6 House He manifold connected and regulator delivery pressure set at 25 psig. ± —
.7 House compressed air operable and line pressure set at 75 psig. ± —
.8 CH_4 cylinders full and regulator delivery pressure set at 40 psig. ± —
.9 Bell jar sight tube N_2 purge rotameter connected and operable. Set rotameter at 2 scfh. C
.10 MTS tank full and Tylan system connected ± —
.11 Tylan control switches SV-1, SV-2, and SV-3 to ~~OPEN~~ 'CLOSED' — —
.12 Open air supply valve to Tylan unit (75 psig) ± —
.13 Open valve to He supply for Tylan unit (25 psig) ± —
2.8 Furnace Out Gas Procedure
2.8.1 Change Test ID number to 16001 (DAS procedure 1.2) — —
.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) — —
.3 Set DAS to scan, record and print at 1 hr intervals. (DAS procedure 1.4) — —

+ Not used in this test

~~NOT SET~~ C

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TABLE D-I (continued)

-5- 9/9/76

2.8.4 Place pyrometers in ~~[illegible]~~ Cal" mode

.5 Initiate DAS scan and record DAS time 13:00:00 Day 264

.6 Close all inlet gas valves

.7 Close outlet valve

.8 Open valve in roughing pump line

.9 Wait until furnace pressure is approx 200 microns, close roughing line valve and open valve to diffusion pump. Provide supply of liquid N_2 for cold trap

.10 Wait until furnace pressure is under $5 \cdot 10^{-4}$ mm Hg

.11 Set DAS to scan at ~~5 min~~ 1 Hr. intervals (DAS procedure 1.4) Print out all data at ~~30 min~~ 1 Hr. intervals. SEE 2.8.3

.12 Initiate DAS scan and record DAS time SEE 2.8.5

.13 Start motor

.14 Turn generator output control to zero

.15 Close generator field contactor

.16 Close generator line contactor

.17 Raise generator output voltage to approx ~~5%~~ 10%

.18 Observe furnace pressure, if it is rising rapidly, reduce the generator voltage; if it is not rising, the voltage may be increased slightly. The objective is to gradually increase the temperature and not let the furnace pressure exceed the range of the 10^{-4} mm Hg scale. ~~Observe gas analyzer. A persistent or rising scan on HASS NO 13 could indicate a water leak inside the furnace. Leaks at HASS NO's 28 and 32 could indicate an air leak.~~ Heating should not proceed until it is determined that any leak indications are not serious. When it has been determined that operating conditions are satisfactory, slowly increase the temperature (T-7) to 1753± 10 °C, keeping the pressure in the 10^{-4} mm Hg range. Observe all water flow, temperature, and the gas analyzer display. Hold the temperature at 1753 ± 10 °C until the furnace pressure is down to approx $5 \cdot 10^{-5}$ mm Hg. Keep power on and go to Power and Flow Tests.

3.0 Power and Flow Tests GO TO PARA 1.10

3.1 N_2 Flow Test #1

3.1.1 Discontinue DAS scan (DAS procedure 1.4.1) Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 08:20:00

SCAN @ 5 MIN PRINT @ 30 MIN .2 Change Test ID number to 16002 (DAS procedure 1.2) Initiate DAS scan and record DAS time Day 265 0855

GO TO PARA 2.8.4 .3 Set N_2 Process flow rate to 23.33 scfm (661.1 l/min).

-6- 9/9/76

3.1.4 Adjust power level to maintain and stabilize (T-7) at 1753 ± 10°C. 13:15 T=1750

.5 Maintain power level for 1 hour with no changes. If T-7 drifts more than 25°C, repeat 3.1.4

.6 Repeat 3.1.1 DAS time 14:26

.7 Exit Gas Temperature Profile.

3.7.1 Verify T-10 thermocouple is at maximum insertion minus ½ inch. This is where it should normally be "parked".

.2 Set DAS for continusous scan of Channels 06-11.

.3 Take continuous scans on DAS for 30 seconds (approx 10 sets of data) Print out the 1st 2 scans of data. Hand record DAS time, thermocouple T-10 position and T-10 output millivolts (DAS channel 09). Discontinue recording on the DAS while the probe is being moved.

.4 Withdraw the T-10 thermocouple ½ inch and repeat the 30 second DAS scan, print out the 1st and 2 nd scans of data, and the recording of time, position, and mV.

.5 Continue the process, each time moving the T-10 thermocouple in ½ inch increments until the diameter of the exit tube (approx ~~[illegible]~~ 3 inches) has been traversed.

DAS time	T-10 position	T-10 output millivolts
14:30:00	PARK	.4068
14:33:00	-½"	.3917
14:36:00	-1"	.3688
14:39:00	-1½"	.3323
14:42:00	-2"	.2877
14:45:00	-2½"	.2174
14:48:00	-3"	.1775

.6 At the end of the traverse, return the T-10 thermocouple to its ~~fully inserted~~ "parking" position ~~minus ½ inch~~.

.7 Repeat 3.1.1

3.2 N_2 Flow Test #2

3.2.1 Change Test ID number to 16003 (DAS procedure 1.2).

.2 Repeat 2.8.2

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TABLE D-I (continued)

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3.2.3 Repeat 2.8.11 JC —

.4 Repeat 2.8.12 DAS time 15:35:00 JC —

.5 Adjust N_2 Process flow rate ~~29.17~~ scfm ~~(1026.4~~ 1/min). 41.67 C1180.1 JC —

.6 Maintain all other parameters including power level — TC

.7 Maintain 29.17 scfm until a stable temperature(T-7), ± 15°C, is achieved for 1 hour. — TC

.8 Repeat 3.1.1 Time 1830 JC —

.9 Repeat 3.1.7

DAS time	T-10 position	T-10 output millivolts
18:33:00	PARK	.2668
18:36:00	-1/2"	.2655
18:39:00	-1"	.2618
18:42:00	-1 1/2"	.2599
18:45:00	-2"	.2379
18:48:00	-2 1/2"	.1967
18:51:00	-3"	.1973

JC —

.10 Repeat 3.1.1 DAS time 18:53 JC —

3.3 Power Test

3.3.1 Change Test ID number to 16004 (DAS procedure 1.2) ± —

.2 Repeat 2.8.2 ± —

.3 Repeat 2.8.11 ± —

.4 Repeat 2.8.12 DAS time _____ ± —

.5 Adjust power level upward to a level 1.25 (kW meter) times that used in 3.2 ± —

.6 Maintain all other parameters, including N_2 flow ± —

.7 Maintain this power level until a stable temperature (T-7), ± 15°C, is achieved for 1 hour. ± —

.8 Repeat 3.1.1 Time _____ ± —

.9 Repeat 3.1.7

DAS time	T-10 position	T-10 output millivolts

± —

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3.3.10 Repeat 3.1.1 Time _____ ± —

4.0 Test Termination

4.1 Change Test ID number to 16005 (DAS procedure 1.2) JC —

4.2 Set DAS to scan at 1 min intervals (DAS procedure 1.4) and print out at 15 min intervals JC —

4.3 Initiate DAS scan and record DAS time 18:58:02 JC —

4.4 Turn High Frequency power off C —

4.5 Maintain N_2 gas flow rate of previous test C —

4.6 After 1 hour turn off Process N_2 flow C —

4.7 Repeat 3.1.1 JC —

4.8 Set Pyrometers to "CAL" mode. Set DAS for continuous scan of channels 00-31 (DAS procedure 1.4) C —

4.9 Initate DAS scan and take 10 scans of data. Printout the first and last scans. JC —

4.10 Cool and inert furnace C —

4.11 Using the tape recorder control, add two (2) file gaps and rewind the tape. Verify tape has been labeled with date, Test ID numbers. Turn off DAS and Tape Deck (DAS procedure 1.4 & 2.2.1). JC —

4.12 Cover pyrometer lenses. JC —

4.13 Turn off all instruments. JC —

(signed) Terry C Wallace
[illegible]
[illegible]
[illegible]
[illegible]
[illegible]

TABLE D-II

POWER AND COATING TEST PROCEDURES

OPERATOR'S copy

LASL COATING FURNACE
POWER AND COATING TEST PROCEDURES
October 1976

1.0 Assembly

1.1 Fixtures assembled as per 26 Y 199115. Check following items for proper location, alignment, and assure that correct transducers are in place.

1.1.1	T-1	Gas-Furnace Inlet	—	
.2	T-2	Bottom Wall Inlet Tube	INOP	
.3	T-3	Middle Wall Inlet Tube	—	
.4	T-4	Top Wall Inlet Tube	—	
.5	T-5	Outside Surf. Carbon Felt	—	
.6	T-6	Back Surf. Substrate	—	
.7	T-7	Deposition Surf. Substrate	—	
.8	T-8	Bottom Wall Exit Tube (Thermowell only)	INOP	
.9	T-9	180° from T-8	—	
.10	T-10	Gas Exhaust (Thermocouple at full insertion minus ½ inch)	—	
.11	T-11	Cooling Water Supply	—	
.12	T-12	Cooling Water Return - Top & Mid Bell Jar Jackets	—	
.13	T-13	Cooling Water Return - Heating Coil Support	—	
.14	T-14	Cooling Water Return - Center Body	—	
.15	T-15	Cooling Water Return - Base Bell Jar	—	
.16	T-16	Cooling Water Return - Precooler Coil	—	
.17	T-17	Cooling Water Return - Heating Coil	—	
.18	T-18	Cooling Water Return - Top Canopy	—	
.19	T-19	Room Temperature	—	
.20	T-20	Cooling Water Return - Capacitor	—	
.21	T-21	Cooling Water Return - Capacitor Bus Bar	—	
.22	F-1	Lower Plenum Pressure	—	
.23	F-1	Mass Flow - Process N_2	—	
.24	F-2	Mass Flow Rate - MTS	—	
.25	F-3	Mass Flow Rate - CH_4	—	
.26	F-4	Mass Flow Rate - He	—	
.27	F-6	Flow Rate - Cooling Water - Top & Mid Bell Jar Jacket	—	
.28	F-7	Flow Rate - Cooling Water - Center Body	INOP	
.29	F-8	Flow Rate - Cooling Water - Heating Coil	—	
.30	F-9	Flow Rate - Cooling Water - Heating Coil Support	—	
1.1.31	F-10	Flow Rate - Cooling Water - Base Bell Jar	—	
.32	F-11	Flow Rate - Cooling Water - Precooler Coil	—	
.33	F-12	Flow Rate - Cooling Water - Top Canopy	—	
.34	W-1	Power (EI Cosϕ)	—	
.35	W-2	Power (EI)	—	
.36	MS-1	Mass Spectrometer	—	

1.2 Cooling water connections to:

1.2.1	Top & Mid Bell Jar Jackets	—	
.2	Heating Coil Support	—	
.3	Center Body	—	
.4	Base Bell Jar	—	
.5	Precooler Coil	—	
.6	Heating Coil	—	
.7	Top Canopy	—	
.8	Capacitor Bus Bar	—	
.9	Capacitor Bus Bar	—	
.10	Power Supply	—	
.11	Mass Spectrometer Mount	—	

1.3 Process gas connections for:

1.3.1	Process N_2	—	
.2	CH_4 (to Tylan Panel)	—	
.3	He (to Tylan Panel)	—	
.4	House N_2 (to Tylan Panel)	—	
.5	MTS	—	
.6	Air (to Tylan Panel for pneumatic valves)	—	

1.4 Initial MTS System Purge

1.4.1	Verify that MTS bubbler tank has been filled.	—	
.2	All valves closed (SV-1 through SV-11)	—	
.3	Remove the line from the Tylan system at the point it connects to the inlet gas valve. Connect a length of hose to the line from the Tylan stytem. Place the other end of the hose inside the facility's exhaust duct.	—	
.4	Turn "ON" the 24 volt power supply	—	
.5	Place the Tylan control knob to "He"	—	
.6	Adjust He pressure to 25 psig. Adjust House N_2 pressure to 25 psig. Adjust CH_4 pressure to ~~25~~ 40 psig.	—	

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TABLE D-II (continued)

1.4.7 Set Valve # SV-4 to "OPEN"
Set Valve # SV-1 to "OPEN"
Set Valve # SV-2 to "OPEN"
Set Valve # SV-6 to "BYPASS"
Set Valve # SV-5 to "BYPASS"

.8 Flow Helium for 5 minutes

.9 Set Valve # SV-3 to "OPEN"

.10 Purge with CH_4 for 5 minutes

.11 Set Valve # SV-3 to "CLOSED"

.12 Continue He purge for 15 minutes

.13 Set Valve # SV-4 to "OFF"
Valve # SV-2 to "CLOSED"
Valve # SV-5 to "OFF"
Valve # SV-6 to "OFF"
Valve # SV-1 to "CLOSED"

1.5 Purge gas connection To T-6 and T-7

1.6 Furnace Power connections

1.7 Capacitor connections made and recorded

1.8 Turn on all instrumentation and power supplies

1.9 Set Test ID number to 17000. (See DAS procedures 2.1 and 1.2) Identify magnetic tape with Test ID number and date. Load tape recorder.

1.10 After 1 hr perform 10 complete data scans. Print out 1st and last scans. (See DAS procedure 1.4) Insert 1 file gap (DAS procedure 2.2.1) on magnetic tape.

2.0 Pre-Heatup Check Out

2.1 Process and House N_2 Supply

2.2 CH_4 Supply

2.3 MIS Supply

2.4 He Supply

2.5 Air Supply

2.6 Water flow rate measurement:

2.6.1 Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-12, F-9, F-6) NOT COMPLETE

.2 Open WR-1

.3 Close WS-2

.4 Open WS-3

.5 Close WS-4

.6 Close WS-5

.7 Open WS-6

2.6.8 Open WS-7

.9 Close WS-8

.10 Close WS-10

.11 Close WS-11

.12 Open WS-9

.13 Open WS-12

.14 Open WS-1

.15 Initiate DAS for continuous scan of above channels and take a minimum of 10 complete scans prior to preceeding to next step. (DAS procedure 1.4) Print out the first two scans of data (DAS procedure)

.16 Record DAS time _____

.17 Discontinue DAS scan (DAS procedure 1.4.1) Insert 1 file gap on magnetic tape (DAS procedure 2.2.1)

.18 Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-11, F-10, F-6)

.19 Close WS-3

.20 Open WS-2

.21 Close WS-6

.22 Open WS-5

.23 Close WS-9

.24 Open WS-8

.25 Close WS-12

.26 Open WS-11

.27 Repeat 2.6.15

.28 Record DAS time _____

.29 Repeat 2.6.17

.30 Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-8, F-7, F-6)

.31 Close WS-5

.32 Open WS-6

.33 Open WS-4

.34 Close WS-11

.35 Open WS-10

.36 Open WS-12

.37 Repeat 2.6.15

.38 Record DAS time _____

.39 Repeat 2.6.17

NOT COMPLETE.

TABLE D-II (continued)

2.7 LASL (SM-29) House Services set up and checked off as follows:

2.7.1 N_2(Process) trailer full — C
.2 CNC-2 service shut off and Group notified. — C
.3 Trailer connected, manifold purged, delivery pressure set at 75 psig. — C
.4 Process N_2 pressure gage at flow meter checked at 80 psig. — C
.5 House N_2 (purge) manifold connected and regulator delivery pressure set at 25 psig. — C
.6 He cylinders full and regulator delivery pressure set at 25 psig. — C
.7 House compressed air operable and line pressure set at 75 psig. — C
.8 CH_4 cylinders full and regulator delivery pressure set at 40 psig. — C
.9 Bell jar sight tube N_2 purge rotameter connected and operable. Set rotameter at 10 scfh. — C
.10 MTS tank full and Tylan system connected — C
.11 Tylan control switches SV-1, SV-2, and SV-3 to "CLOSED" position. — C
.12 Open air supply valve to Tylan unit (75 psig) -- C
.13 Open valve to He supply for Tylan unit (25 psig) — C

2.8 Furnace Out Gas Procedure

2.8.1 Change Test ID number to 17001 (DAS procedure 1.2) Not Done QC
.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) NO —
.3 Set DAS to scan, record and print at 1 hr intervals. (DAS procedure 1.4) NO —
.4 Initiate DAS scan and record DAS time _____ NO
.5 Close all inlet gas valves — C
.6 Close outlet valve — C
.7 Open valve in roughing pump line — C
.8 Wait until furnace pressure is approx 200 microns, close roughing line valve and open valve to diffusion pump. Provide supply of liquid N_2 for cold trap — C
.9 Wait until furnace pressure is under $5 \cdot 10^{-4}$ mm Hg — C
Record DAS time Done Panels

2.8.10 Start motor — C
.11 Turn generator output control to zero — C
.12 Close generator field contactor — C
.13 Close generator line contactor — C
.14 Raise generator output voltage to approx 80 40 V. — C
.15 Place pyrometers in "RUN" mode — C
.16 Observe furnace pressure, if it is rising rapidly, reduce the generator voltage; if it is not rising, the voltage may be increased slightly. The objective is to gradually increase the temperature and not let the furnace pressure exceed the range of the 10^{-4} mm Hg scale. Observe gas analyzer. A persistent or rising peak at MASS NO 18 could indicate a water leak inside the furnace. Peaks at MASS NO's 28 and 32 could indicate an air leak. Heating should not proceed until it is determined that any leak indications are not serious. When it has been determined that operating conditions are satisfactory, slowly increase the temperature (T-7) to 1350 ± 10 °C, keeping the pressure in the 10^{-4} mm Hg range. Observe all water flow, temperature, and the gas analyzer display. Hold the temperature at 1350 ± 10 °C until the furnace pressure is down to approx $5 \cdot 10^{-4}$ mm Hg. Keep power on and go to Power Tests. — C

3.0 Power Tests
3.1 Power Test #1

3.1.1 Discontinue DAS scan (DAS procedure 1.4.1) Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 162500.
.2 Set DAS to scan at 5 min intervals (DAS procedure 1.4) Print out all data at 30 min intervals. QC —
.3 Change Test ID number to 17002 (DAS procedure 1.2) Initiate DAS scan and record DAS time 162500 QC —
.4 Set N_2 Process flow rate to 16.67 scfm (472.3 l/min) QC —
.5 Adjust power level to maintain and stabilize (T-7) at 1350 ± 10°C — C
.6 Maintain power level for 1 hour with no changes. If T-7 drifts more than 25°C, repeat 3.1.5 — C
.7 Repeat 3.1.1 QC —

3.2 Power Test #2.

3.2.1 Change Test ID number to 17003 (DAS procedure 1.2) QC —
.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) QC —
.3 Set DAS to scan at one min. intervals. Print at 5 min. intervals. (DAS procedure 1.4). QC —

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TABLE D-II (continued)

3.2.4 Initiate DAS scan and record DAS time 205000 QC
.5 Adjust power level upward to a level 1.5 (kW meter) times that used in 3.1.5. C
.6 Maintain all other parameters, including N_2 flow. C
.7 Maintain this power level until a stable temperature (T-7), ± 15°C, is achieved for 1 hour. C
.8 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 225130. QC

3.3 Set-up Coating Conditions
3.3.1 Change TEST ID number to 17004 (DAS procedure 1.2) QC
.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) QC
.3 Set DAS to scan, record and print at 5 min intervals (DAS procedure 1.4) QC
.4 Initiate DAS scan and record DAS time 229500 QC
.5 Raise N_2 Process flow rate to 30.00 scfm (849.5 l/min) QC
.6 Adjust power to maintain 1753 ± 10°C (T-7). Record DAS time at each adjustment of power. LD
.7 Stabilize substrate inner surface temperature at 1753 ± 10°C (T-7) for at least one hour with no adjustments greater than ± 2 kW/15 min. LD
.8 Record time stabilization is achieved 04 22 DAS time 03 - LD
.9 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 00 40 00. LD

4.0 Coating Runs
4.1 Coating Run #1
4.1.1 Change Test ID number to 17005 (DAS procedure 1.2) LD
.2 Set DAS to scan channels 00-31 (DAS procedure 1.4). LD
.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4). LD
.4 Initiate DAS scan and record DAS time 20 50 00 LD
.5 Pyrographite release layer. Set CH_4 flow controller to 8.58 SLPM (Tylan Vernier - 8.60) Turn Tylan switch SV-3 (CH_4) to "ON" Record DAS time 2055 17 LD
.6 After 10 minutes set MTS flow controller to 8.94 g/min (Tylan Vernier - 9.93) LD
.7 Turn Tylan switch SV-1 to "ON" (MTS) Record DAS time 21 00 00 Pr. T.D. LD
.8 Adjust Power as required to maintain T-7 at 1753 ± 10°C. Record DAS time at each adjustment of power. LD

4.1.9 After 4 hours from 4.1.7, discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 04 32 00 LD

4.2 Set-up Coating Run #2
4.2.1 Change TEST ID number to 17006. LD
.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) LD
.3 Set DAS to scan, record and print at 5 min intervals (DAS procedure 1.4). LD
.4 Initiate DAS scan and record DAS time 05 00 00 LD
.5 Turn Tylan Switch SV-1 to "OFF"(MTS) Record DAS time 04 32 47 LD
.6 Turn Tylan Switch SV-3 (CH_4) to "OFF". Record DAS time 04 33 00 LD
.7 Set N_2 Process Flow rate to 23.33 scfm (660.7 l/min) Record DAS time 04 54 09 LD
.8 Adjust Power to maintain 1753 ± 10°C (T-7). Record DAS time at each adjustment of power. QC
.9 Stabilize substrate inner surface temperature at 1753 ± 10°C (T-7) for at least 1 hour from 4.2.8 with no adjustment in power required greater than ± 2 kW/15 min. QC
.10 Record time stabilization is achieved 0530 QC
.11 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 0532. QC

4.3 Coating Run #2
4.3.1 Change TEST ID number 17007 LC
.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) QC
.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4). QC
.4 Initiate DAS scan and record DAS time 0535 QC
.5 Pyrographite release layer. Set CH_4 flow controller to 6.67 SLPM (Tylan Vernier - 6.69). Turn Tylan Switch SV-3 (CH_4) to "ON". Record DAS time 0538 QC
.6 After 10 minutes set MTS flow controller to 6.94 g/min (Tylan Vernier - 7.71) QC
.7 Turn Tylan Switch SV-1 to "ON" (MTS) Record DAS time 0545 QC
.8 Adjust power as required to maintain T-7 at 1753 ± 10°C. Record DAS time at each adjustment of power. QC
.9 After 4 hours from 4.3.7 discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 1246 QC

TABLE D-II (continued)

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4.4 Set-up Coating Run #3

4.4.1 Change TEST ID number to 17008 — QC —

.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) — QC —

.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4). — QC —

.4 Initiate DAS scan and record DAS time 125000 — QC —

.5 Turn Tylan Switch SV-1 to "OFF"(MFS) — QC —

.6 Turn Tylan Switch SV-3 to "OFF"(CH_4) — QC —

.7 Set N_2 Process Flow rate to 16.67 scfm (471.9 l/min). Record DAS time 1420 — QC —

.8 Adjust Power to maintain 1753 ± 10°C (T-7). Record DAS time at each adjustment of power. — QC —

.9 Stabilize substrate inner surface temperature at 1753 ± 10°C (T-7) for at least 1 hour from 4.4.8 with no adjustment in power required greater than ± 2 kW/15 min. — QC —

.10 Record time stabilization is achieved 1455 — QC —

.11 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 1455. — QC —

4.5 Coating Run #3

4.5.1 Change Test ID number to 17009 — QC —

.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) — QC —

.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4) — QC —

.4 Initiate DAS scan and record DAS time 1455 — QC —

.5 Pyrographite release layer. Set CH_4 flow controller to 4.77 SLPM (Tylan Vernier - 4.79). Turn Tylan Switch SV-3 (CH_4) to "ON". Record DAS time 155540 — QC —

.6 After 10 minutes set MFS flow controller to 4.94 g/min (Tylan Vernier - 5.49) — QC —

.7 Turn Tylan Switch SV-1 to "ON" (MFS) Record DAS time 160140 — QC —

.8 Adjust power as required to maintain T-7 at 1753 ± 10°C Record DAS time at each adjustment of power. — QC —

.9 After 4 hours from 4.5.7, discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 170100 — QC —

5.0 Test Termination

5.1 Change TEST ID number to 17010 — QC —

.2 Set DAS to scan channgel 00-31 (DAS procedure (1.4) — QC —

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5.3 Set DAS to scan at 1 min intervals (DAS procedure 1.4) and print out at 15 min intervals. — QC —

5.4 Initiate DAS scan and record DAS time 170201 — QC —

5.5 Set Tylan Switch SV-1 to "OFF" (MFS) 170252 — QC —

5.6 Set Tylan Switch SV-3 to "OFF" (CH_4) 170256 — QC —

5.7 Turn High Frequency Power off. Record DAS time 170330 — QC —

5.8 Adjust Process N_2 Flow to 3.3 SCFM (94.45 l/min) Record DAS time 170900 — QC —

5.9 After 1 hour from 5.3.7, Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1). Record DAS time 180930 — QC —

5.10 Set Pyrometers to "CAL" mode. Set DAS for continuous scan of channels 00-31 (DAS procedure 1.4) — QC —

PURGE TYLAN SYSTEM

5.11 Initiate DAS scan and take 10 scans of data. Printout the first and last scans. — QC —

5.12 Cool and inert furnace — QC —

5.13 Using the tape recorder control, add two (2) file gaps and rewind the tape. Verify tape has been labled with date & TEST ID numbers, Turn off DAS and Tape Deck (DAS procedure 1.4 & 2.2.1) — QC —

5.14 Cover Pyrometer lenses. — QC —

5.15 Purge Tylan MFS System

5.15.1 Set Valve SV-5 to "BYPASS" — QC —

.2 Set Valve SV-6 to "BYPASS" — QC —

.3 Set Valve SV-1 to "OPEN" — QC —

.4 Set Valve SV-2 to "OPEN" — QC —

.5 Purge with He for 1 hour — QC —

.6 Set Valve SV-4 to "OFF" — QC —

.7 Set Valve SV-1 to "CLOSED" — QC —

.8 Set Valve SV-2 to "CLOSED" — QC —

.9 Set Valve SV-5 to "OFF" — QC —

.10 Set Valve SV-6 to "OFF" — QC —

5.16 Turn off all instruments — QC —

(signed) J. C. M. H.
G. V. Sampson
W. C. Dalton
Dale Crook
C. Wald

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TABLE D-III

SECOND COATING RUN TEST PROCEDURES

Master

LASL COATING FURNACE
2nd COATING RUN TEST PROCEDURES
NOVEMBER 1976

1.0 Assembly

1.1 Fixtures assembled as per 26 Y 199115. Check following items for proper location, alignment, and assure that correct transducers are in place.

1.1.1	T-1	Gas-Furnace Inlet	C	—
.2	T-2	Bottom Wall Inlet Tube	C	—
.3	T-3	Middle Wall Inlet Tube	C	—
.4	T-4	Top Wall Inlet Tube	C	—
.5	T-5	Outside Surf. Carbon Felt	C	—
.6	T-6	Back Surf. Substrate	C	—
.7	T-7	Deposition Surf. Substrate	C	—
.8	T-8	Bottom Wall Exit Tube (~~Thermowell only~~)	X	~~N/A~~
.9	T-9	180° from T-8	C	—
.10	T-10	Gas Exhaust (Thermocouple at full insertion minus ½ inch)	C	—
.11	T-11	Cooling Water Supply	C	—
.12	T-12	Cooling Water Return - Top & Mid Bell Jar Jackets	C	—
.13	T-13	Cooling Water Return - Heating Coil Support	C	—
.14	T-14	Cooling Water Return - Center Body	C	—
.15	T-15	Cooling Water Return - Base Bell Jar	C	—
.16	T-16	Cooling Water Return - Precooler Coil	C	—
.17	T-17	Cooling Water Return - Heating Coil	C	—
.18	T-18	Cooling Water Return - Top Canopy	C	—
.19	T-19	Room Temperature	C	—
.20	T-20	Cooling Water Return - Capacitor	C	—
.21	T-21	Cooling Water Return - Capacitor Bus Bar	C	—
.22	P-1	Lower Plenum Pressure	C	—
.23	F-1	Mass Flow - Process N_2	C	—
.24	F-2	Mass Flow Rate - MTS	C	—
.25	F-3	Mass Flow Rate - CH_4	C	—
.26	F-4	Mass Flow Rate - He	C	—
.27	F-6	Flow Rate - Cooling Water - Top & Mid Bell Jar Jacket	C	—
.28	F-7	Flow Rate - Cooling Water - Center Body	C	—
.29	F-8	Flow Rate - Cooling Water - Heating Coil	C	—
.30	F-9	Flow Rate - Cooling Water - Heating Coil Support	C	—

TABLE D-III (continued)

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1.1.31	F-10 Flow Rate - Cooling Water - Base Bell Jar	C —
.32	F-11 Flow Rate - Cooling Water - Precooler Coil	C —
.33	F-12 Flow Rate - Cooling Water - Top Canopy	C —
.34	W-1 Power (EI Cos∅)	C —
.35	W-2 Power (EI)	C —
.36	MS-1 Mass Spectrometer AUX.	C —
.37	F-13 CH_4 Cross Flow RATE	C —
1.2	Cooling water connections to:	C —
1.2.1	Top & Mid Bell Jar Jackets	C —
.2	Heating Coil Support	C —
.3	Center Body	C —
.4	Base Bell Jar	C —
.5	Precooler Coil	C —
.6	Heating Coil	C —
.7	Top Canopy	C —
.8	Capacitor Bus Bar	C —
.9	Capacitor Bus Bar	C —
.10	Power Supply	C —
.11	Mass Spectrometer Mount	C —
1.3	Process gas connections for:	
1.3.1	Process N_2	C —
.2	CH_4 (to Tylan Panel)	C —
.3	He (to Tylan Panel)	C —
.4	House N_2 (to Tylan Panel)	C —
.5	MTS	C —
.6	Air (to Tylan Panel for pneumatic valves)	C —
1.4	Initial MTS System Purge	— —
1.4.1	Verify that MTS bubbler tank has been filled.	— CMH
.2	All valves closed (SV-1 through SV-11)	— CMH
.3	Remove the line from the Tylan system at the point it connects to the inlet gas valve. Connect a length of hose to the line from the Tylan stytem. Place the other end of the hose inside the facility's exhaust duct.	— N/A
.4	Turn "ON" the 24 volt power supply	— CMH
.5	Place the Tylan control knob to "He"	— CMH
.6	Adjust He pressure to 25 psig. Adjust House N_2 pressure to 25 psig. Adjust CH_4 pressure to 25 psig.	— CMH

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1.4.7	Set Valve # SV-4 to "OPEN"	— CMH
	Set Valve # SV-1 to "OPEN"	— CMH
	Set Valve # SV-2 to "OPEN"	— CMH
	Set Valve # SV-6 to "BYPASS"	— CMH
	Set Valve # SV-5 to "BYPASS"	— CMH
.8	Flow Helium for 5 minutes	— CMH
.9	Set Valve # SV-3 to "OPEN"	— CMH
.10	Purge with CH_4 for 5 minutes	— CMH
.11	Set Valve # SV-3 to "CLOSED"	— CMH
.12	Continue He purge for 15 minutes	— CMH
.13	Set Valve # SV-4 to "OFF"	
	Valve # SV-2 to "CLOSED"	
	Valve # SV-5 to "OFF"	
	Valve # SV-6 to "OFF"	
	Valve # SV-1 to "CLOSED"	— CMH
1.5	Purge gas connection To T-6 and T-7	C —
1.6	Furnace Power connections	C —
1.7	Capacitor connections made and recorded	C —
1.8	Turn on all instrumentation and power supplies	— JP
1.9	Set Test ID number to 10000. (See DAS procedures 2.1 and 1.2) Identify magnetic tape with Test ID number and date. Load tape recorder.	— JP
1.10	After 1 hr perform 10 complete data scans. Print out list and last scans. (See DAS procedure 1.4) Insert 1 file gap (DAS procedure 2.2.1) on magnetic tape.	— JP
2.0	Pre-Heatup Check Out	
2.1	Process and House N_2 Supply	C —
2.2	CH_4 Supply	C —
2.3	MTS Supply	C —
2.4	He Supply	C —
2.5	Air Supply	C —
2.6	Water flow rate measurement:	C —
2.6.1	Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-12, F-9, F-6)	N/A —
.2	Open WR-1	—
.3	Close WS-2	—
.4	Open WS-3	—
.5	Close WS-4	—
.6	Close WS-5	—
.7	Open WS-6	N/A —

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TABLE D-III (continued)

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2.6.8	Open WS-7	N/A
.9	Close WS-8	
.10	Close WS-10	
.11	Close WS-11	
.12	Open WS-9	
.13	Open WS-12	
.14	Open WS-1	
.15	Initiate DAS for continuous scan of above channels and take a minimum of 10 complete scans prior to preceeding to next step. (DAS procedure 1.4) Print out the first two scans of data (DAS procedure)	
.16	Record DAS time _____	
.17	Discontinue DAS scan (DAS procedure 1.4.1) Insert 1 file gap on magnetic tape (DAS procedure 2.2.1)	
.18	Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-11, F-10, F-6)	
.19	Close WS-3	
.20	Open WS-2	
.21	Close WS-6	
.22	Open WS-5	
.23	Close WS-9	
.24	Open WS-8	
.25	Close WS-12	
.26	Open WS-11	
.27	Repeat 2.6.15	
.28	Record DAS time _____	
.29	Repeat 2.6.17	
.30	Set DAS to scan channels 00-03 (DAS procedure 1.4) (DAS Ref, F-8, F-7, F-6)	
.31	Close WS-5	
.32	Open WS-6	
.33	Open WS-4	
.34	Close WS-11	
.35	Open WS-10	
.36	Open WS-12	
.37	Repeat 2.6.15	
.38	Record DAS time _____	
.39	Repeat 2.6.17	N/A

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2.7	LASL (SM-29) House Services set up and checked off as follows:	
2.7.1	N_2(Process) trailer full	C
.2	CNC-2 service shut off and Group notified.	C
.3	Trailer connected, manifold purged, delivery pressure set at 75 psig.	C
.4	Process N_2 pressure gage at flow meter checked at 80 psig.	C
.5	House N_2 (purge) manifold connected and regulator delivery pressure set at 25 psig.	C
.6	He cylinders full and regulator delivery pressure set at 25 psig.	C
.7	House compressed air operable and line pressure set at 75 psig.	C
.8	CH_4 cylinders full and regulator delivery pressure set at 40 psig.	C
.9	Bell jar sight tube N_2 purge rotameter connected and operable. Set rotameter at 5 scfh.	
.10	MTS tank full and Tylan system connected	C
.11	Tylan control switches SV-1, SV-2, and SV-3 to "CLOSED" position.	
.12	Open air supply valve to Tylan unit (75 psig)	C
.13	Open valve to He supply for Tylan unit (25 psig)	C
2.8	Furnace Out Gas Procedure	
2.8.1	Change Test ID number to 13001 (DAS procedure 1.2)	N/A
.2	Set DAS to scan channels 00-31 (DAS procedure 1.4)	
.3	Set DAS to scan, record and print at 1 hr intervals. (DAS procedure 1.4)	
.4	Initiate DAS scan and record DAS time _____	N/A
.5	Close all inlet gas valves	C
.6	Close outlet valve	C
.7	Open valve in roughing pump line	C
.8	Wait until furnace pressure is approx 200 microns, close roughing line valve and open valve to diffusion pump. Provide supply of liquid N_2 for cold trap	
.9	Wait until furnace pressure is under $5 \cdot 10^{-4}$ mm Hg	C
	~~Record DAS time~~ _____	

TABLE D-III (continued)

2.8.10 Start motor

.11 Turn generator output control to zero

.12 Close generator field contactor

.13 Close generator line contactor

.14 Raise generator output voltage to approx 40 V.

.15 Place pyrometers in "RUN" mode

.16 Observe furnace pressure, if it is rising rapidly, reduce the generator voltage; if it is not rising, the voltage may be increased slightly. The objective is to gradually increase the temperature and not let the furnace pressure exceed the range of the 10^{-4} mm Hg scale. Observe gas analyzer. A persistent or rising peak at MASS NO 18 could indicate a water leak inside the furnace. Peaks at MASS NO's 28 and 32 could indicate an air leak. Heating should not proceed until it is determined that any leak indications are not serious. When it has been determined that operating conditions are satisfactory, slowly increase the temperature (T-7) to 900 ± 10 °C, keeping the pressure in the 10^{-4} mm Hg range. Observe all water flow, temperature, and the gas analyzer display. Hold the temperature at 900 ± 10 °C until the furnace pressure is down to approx $5 \cdot 10^{-4}$ mm Hg.

2.9 Overnight Cool Down.

.1 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1). Record DAS time 15:57:11 DAY 310

.2 Turn High Frequency Power off.

.3 Set pyrometers to "SIL" mode. Set DAS for continuous scan of channels 00-31 (DAS procedure 1.4)

.4 Initiate DAS scan and take 10 scans of data. Print out the first and last scans.

.5 Turn off all instruments.

.6 Cover pyrometer lenses

.7 Leave connection between vacuum pumps and bell jar "OPEN" overnight.

3.0 Mass Spectrometer calibration

3.1 Turn on all instrumentation and power supplies

3.2 Set Test ID number to 18002 (See DAS procedures 2.1 and 1.2)

3.3 Set DAS to scan, record and print at 5 minute intervals. (DAS procedure 1.4)

3.4 "DELETED"

3.5 Initiate DAS scan and record DAS time 10116 DAY 313

3.6 Set H_2 Process flow rate to 3.53 scfh (100 l/min)

3.7 Allow flow system to equilibrate for 5 minutes.

3.8 Set Mass Spectrometer parameters: Ionization current 2.2 ma, Mass range 10 to 100, Sensitivity 10^{-11} amp/div, scan rate 6.800 sec. Record pressure 1.1×10^{-7}

3.9 Set CH_4 flow controller to 9.00 SLPM (Tylan Vernier - 9.02). Turn Tylan switch S7-3 (CH_4) to "ON". Record DAS time 14:30:00 Allow system to equilibrate for 5 minutes.

3.10 Adjust recorder so that Mass 16 peak is full scale. Take trace of spectrum and identify graph CH_4 - 9v/o 9.19%

3.11 Turn CH_4 flow controller to 6.00 (Tylan Vernier -6.02). Record DAS time 14:36 . Allow system to equilibrate for 5 min. 6.3%

3.12 Take mass spectrum trace as above with recorder controls the same. Identify graph as CH_4 -6v/o.

3.13 Turn CH_4 flow controller to 3.00 SLPM (Tylan Vernier - 3.02). Record DAS time 15:12 . Allow system to equilibrate for 5 min.

3.14 Take mass spectrum trace as above with recorder controls the same. Identify graph as CH_4 - 3v/o 3.57%

3.15 Turn CH_4 flow controller 1.00 SLPM (Tylan Vernier 1.02). Record DAS time 15:21 . Allow system to equilibrate for 5 min.

3.16 Take mass spectrum trace as above with recorder controls the same. Identify graph as CH_4 - 1v/o. 1.7%

3.17 Turn Tylan switch S7-3 (CH_4) to "OFF". Record DAS time 15:27 Allow system to equilibrate for 5 min.

3.18 Take mass spectrum trace as above with recorder controls the same. Identify graph as CH_4 -0v/o.

3.19 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1). Record DAS time 16:06 1%

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TABLE D-III (continued)

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4.0 Coating Runs

4.1 Set-up Coating Run 1.

4.1.1 Change TEST ID number to 18003 (DAS procedure 1.2)

.2 Set DAS to scan channels 00-31 (DAS procedure 1.4)

.3 Set DAS to scan, record and print at 5 min intervals.

.4 Initiate DAS scan and record DAS time 16:20:00

.5 Raise H_2 Process flow rate to 10.00 scfm (283.17 l/min)

.6 Turn power to coil "ON"

.7 Adjust power to bring substrate to 1650 ± 10°C (T-7). Record DAS time at each adjustment of power.

.8 Stabilize substrate inner surface temperature at 1650° ± 10°C (T-7) for at least one hour with no adjustments greater than ± 2 kW/15 min.

.9 Record time stabilization is achieved. DAS time 20:05

.10 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 20:07.

4.2 Coating Run #1

4.2.1 Change Test ID number to 18004 (DAS procedure 1.2)

.2 Set DAS to scan channels 00-31 (DAS procedure 1.4).

.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4).

.4 Initiate DAS scan and record DAS time 20:10:00

.5 After 5 min take mass spectrum trace as above with recorder controls the same. Identify graph as start of Run 1 (CH_4).

.6 Pyrographite release layer. Set CH_4 flow controller to 9.00 SLPM (Tylan Vernier - 9.02). Turn Tylan switch SV-3 (CH_4) to "ON"

.7 Turn auxiliary CH_4 source "ON" and adjust at 9.00 SLPM Record DAS time 20:27

.8 After 5 min take mass spectrum trace as above with recorder controls the same. Identify graph as Start of Run 1. (CH_4)

.9 After 15 minutes from 4.2.7 set MTS flow controller to 8.9% g/min (Tylan Vernier - 7.93)

.10 Turn Tylan switch SV-3 to "ON" (MTS). Record DAS time 20:47

.11 Adjust power as required to maintain T-7 at 1650 ± 10°C. Record DAS time at each adjustment of power.

.12 After 5 min take mass spectrum trace as above with recorder controls the same. Identify graph as Start of Run 1 (CH_4 & MTS)

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4.2.13 Take mass spectrum trace every 1 hr. After 4 hours from 4.2.10, discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 01:01:00

4.3 Set-up Coating Run 2

4.3.1 Change TEST ID number to 18005.

.2 Set DAS to scan channels 00-31 (DAS procedure 1.4)

.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4).

.4 Initiate DAS scan and record DAS time 01:02:11

.5 Turn Tylan Switch SV-1 to "OFF"(MTS) Record DAS time 01:02:37

.6 Turn Auxilliary CH_4 source "OFF". Record DAS time 01:03:05

.7 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 01:05:00

4.4 Coating Run 2

4.4.1 Change TEST ID number to 18006

.2 Set DAS to scan, channels 00-31. (DAS procedure 1.4).

.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4).

.4 Initiate DAS scan and record DAS time 01:09:00

.5 5 minutes from 4.3.6 take a mass spectrum trace as above with recorder controls the same. Identify graph as start of run 2. Ramp up Power

.6 After 15 minutes from 4.3.5 set MTS flow controller to 8.9% g/min (Tylan vernier - 9.93)

.7 Turn Tylan switch SV-1 to "ON" (MTS). Record DAS time 02:15:20

.8 5 minutes after 4.4.7, take a mass spectrum trace as above with recorder controls the same. Identify by DAS time. 02:15:20

.9 Adjust power as required to maintain T-7 at 1650 ± 10°C. Record DAS time at each adjustment of power.

.10 Take mass spectrum trace every hour. After 4 hours from 4.4.7, discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) DAS time 02:15:40

TABLE D-III (continued)

4.5 Set-up Coating Run #3

4.5.1 Change TEST ID number to 18007 — —

.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) — —

.3 Set DAS to scan, record and print at 5 min intervals (DAS procedure 1.4). — —

.4 Initiate DAS scan and record DAS time 06:20:00 — —

.5 Turn Tylan Switch SV-1 to "OFF"(MTS) 06:17:00 — —

.6 Turn Tylan Switch SV-3 to "OFF"(CH_4) 06:17:00 — —

.7 Set N_2 Process Flow rate to 16.67 scfm (471.9 l/min). Record DAS time 06:26:00 — —

.8 Adjust Power to maintain 1650 ± 10°C (T-7). Record DAS time at each adjustment of power. — —

.9 Stabilize substrate inner surface temperature at 1650 ± 10°C (T-7) for at least 1 hour from 4.6.8 with no adjustment in power required greater than ± 2 kW/15 min. — —

.10 Record time stabilization is achieved 06:28:00 — —

.11 Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 06:32:00. — —

4.6 Coating Run #3

4.6.1 Change Test ID number to 18008 — —

.2 Set DAS to scan channels 00-31 (DAS procedure 1.4) — —

.3 Set DAS to scan, record and print at 5 min intervals. (DAS procedure 1.4) — —

.4 Initiate DAS scan and record DAS time 06:36:00 — —

.5 After 3 min take mass spectrum trace as above with recorder controls the same. Set CH_4 flow controller to 9.00 SLPM (Tylan Vernier - 9.02). Turn Tylan Switch SV-3 (CH_4) to "ON". Record DAS time 06:43:00 — —

.6 Turn auxiliary CH_4 source "ON" and adjust at 9.00 SPLM Record DAS time 06:41:00 — —

.7 After 5 min take mass spectrum trace as above with recorder controls the same. Identify graph as Start of Run 3 by DAS Time — —

.8 After 15 minutes from 4.6.6 set MTS flow controller to 8.94 g/min (Tylan Vernier - 9.93) — —

.9 Turn Tylan Switch SV-1 to "ON" (MTS) Record DAS time 06:53:00 — —

5 min after 4.6.9, take mass spec. Identify by DAS Time

.10 Adjust power as required to maintain T-7 at 1650 ± 10°C Record DAS time at each adjustment of power. Run out of hours — —

.11 Take mass spectrum trace every 1 hr. After 4 hours from 4.6.9, discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1) Record DAS time 10:36:00 — —

5.0 Test Termination

5.1 Change TEST ID number to 18009 — —

5.2 Set DAS to scan channel 00-31 (DAS procedure (1.4) — —

5.3 Set DAS to scan at 1 min intervals (DAS procedure 1.4) and print out at 15 min intervals. — —

5.4 Initiate DAS scan and record DAS time 10:57:00 — —

5.5 Set Tylan Switch SV-1 to "OFF" (MTS) 10:57:30 — —

5.6 Set Tylan Switch SV-3 to "OFF" (CH_4) and Aux. CH_4 to "OFF" 10:57:30 — —

5.7 Turn High Frequency Power off. Record DAS time 10:51:15

5.8 Adjust process N_2 flow rate to 10 scfm (283.17 l/m) Record DAS time — —

5.9 5 minutes from 5.8 take a mass spectrum trace as above with recorder controls the same. Identify graph as test termination — —

5.10 Adjust Process N_2 Flow to 3.3 SCFM (94.45 l/min) Record DAS time 10:55:11 — —

5.11 After 30 min. from 5.7. Discontinue DAS scan (DAS procedure 1.4.1). Insert 1 file gap on magnetic tape (DAS procedure 2.2.1). Record DAS time 11:45:00 — —

5.12 Set Pyrometers to "CAL" mode. Set DAS for continuous scan of channels 00-31 (DAS procedure 1.4) — —

5.13 Initiate DAS scan and take 10 scans of data. Printout the first and last scans. — —

5.14 Cool and inert furnace — —

5.15 Using the tape recorder control, add two (2) file gaps and rewind the tape. Verify tape has been labled with date & TEST ID numbers. Turn off DAS and Tape Deck (DAS procedure 1.4 & 2.2.1) — —

5.16 Cover Pyrometer lenses. — —

5.17 Purge Tylan MTS System

5.15.1 Set Valve SV-5 to "BYPASS" — —

.2 Set Valve SV-6 to "BYPASS" — —

.3 Set Valve SV-1 to "OPEN" — —

.4 Set Valve SV-2 to "OPEN" — —

.5 Purge with He for 1 hour — —

.6 Set Valve SV-4 to "OFF" — —

.7 Set Valve SV-1 to "CLOSED" — —

.8 Set Valve SV-2 to "CLOSED" — —

.9 Set Valve SV-5 to "OFF" — —

.10 Set Valve SV-6 to "OFF" — —

5.18 Turn off all instruments — —

(signed)

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APPENDIX E
TEST DATA

I. GENERAL

This appendix contains the test logs and graphs of reduced data from the nitrogen flow, power, and deposition tests, with a discussion of data anomalies.

II. NITROGEN FLOW AND POWER TESTS

The test identification numbers for these tests are in the 16000 series. The data are from tests 16002, 16003, and 16005.

B. Test log - Nitrogen flow and power tests.

The original of enclosed test log for test series 16000 is on file at LASL. It is included as part of the data package because it outlines pertinent events and their time of occurence, and so that the reader may correlate these events with the data graphs.

C. Test Data - Nitrogen flow and power tests.

The data that follow depict the part of the test in which power was applied to the furnace. It was not convenient to present the data taken during other parts of the test in this format, so parts of the data are shown in Sec III of this report. All data were recorded on magnetic tape, which is on file at LASL.

D. Discussion of Nitrogen flow and power tests data.

Each data point is illustrated by a plus sign. These points are connected by a straight line. The small arrows on the abscissa depict a different test number. To facilitate data interpretation, the test I.D. numbers are as follows:

	Test I.D. Number	Test Description
(1.)	16002	N_2 Flow Test No. 1
(2.)	16003	N_2 Flow Test No. 2
(3.)	16005	Test Termination

The nitrogen flow rate (parameter F-1) began to fluctuate at 1300 during test 16002. It stabilized again at 1405 h. During this period, it varied from a high of approximately 674 ℓ/min to a low of 632 ℓ/min. This variation was apparently caused by a faulty nitrogen pressure regulator. The regulator became unstable again as the flow rate was being increased to 1180 ℓ/min. The data shows this instability from 1535 to 1635 h. Between 1635 and 1735 h the

NITROGEN FLOW AND POWER TEST LOG

SEPTEMBER 20, 1976 - DAY 264 - TEST 16000 → 05

TIME			
10:36:00	COMPLETED	PARA 2.6.1 thru 2.6.1.15	F/G
10:39:00	COMPLETED	PARA 2.6.1.18 thru 2.6.1.28	F/G
10:42:00	COMPLETED	PARA 2.6.1.30 thru 2.6.1.38	F/G
11:00:00	MADE 1 SCAN ALL CHANNELS		F/G
12:00:00	TEST I.D.to #16001		
	START 1 HOUR SCANS		
13:08:30	FURNACE POWER TURNED ON		
13:30:00	MADE 10 DATA SCANS		
14:00:00	START 1 HOUR SCANS		

SEPTEMBER 21, 1976 - DAY 265 - TEST 16000 → 05

08:15:00	MADE 10 DATA SCANS		F/G
08:20:00	TEST I.D. to 16002		
08:55:00	START 5 MIN DATA SCANS		
09:00:00	DATA SCAN MISSED DURING N_2		
	FLOW ADJUSTMENT		
09:05:00	RESUMED 5 MIN SCANS		
09:06:00	SWITCHED T6 & T7 PYROMETERS		
	TO CAL OFF		
09:10:30	FURNACE POWER ADJUSTMENT		

NOTE: FURNACE TEMPERATURE INCREASING. HAVE N_2 FLOW RATHER THAN VACUUM. N_2 IN FURNACE AT APPROXIMATELY ATMOSPHERIC PRESSURE AND 661.1 SLPM FLOW RATE.

FLOW AND POWER TEST LOG (cont'd)

09:57:00 thru		
11:35:00	MADE SEVERAL INCREASES IN FURNACE POWER LEVEL	
11:51:00	TURNED FURNACE POWER OFF AND ADDED CAPACITANCE TO CIRCUIT. TURNED FURNACE POWER BACK ON AND NOTED CHANGE IN KVAR READING FROM -4 DIVISIONS TO +4.5 DIVISIONS ON METER.	
12:15:00	F-6 FLOWMETER FAILED - NO OUTPUT FROM TURBINE PICKUP	
12:46:00	POWER INCREASED	
12:53:00	NOTICED FOGGING ON INSIDE OF T-6 SIGHT GLASS AND INSULATION SEPARATION	
12:57:00	POWER INCREASED	
13:05:00	POWER INCREASED	
13:07:00	POWER INCREASED	
13:15:00	STARTED PARA. 3.1.4 - STABILIZATION CHECKS	
13:50:00	MADE SMALL INCREASE IN POWER	
14:26:00	DISCONTINUED DAS SCAN-PARA 3.1.6	F/G
14:48:00	COMPLETED PARA 3.1.7.6	F/G
14:55:00	STARTED 5 MIN SCANS	
NOTE: T-4 SHOWED OPEN ON FIRST SCAN		
14:56:00	T-3 and T-4 BOTH INDICATE OPEN	
NOTE: ADDED GROUND ON COMMON TERMINAL OF ±15 VOLT POWER SUPPLY TO DAS GROUND - APPEARS TO HAVE CURED PROBLEM. ALL TEMPERATURES NOW IN NORMAL RANGE.		
15:30:30	TERMINATED 5 MIN SCAN	
	COMPLETED TEST 16002	F/G
15:32:00	TEST I.D. TO 16003	
15:35:00	STARTED 5 MIN SCANS	
15:39:00	N_2 FLOW ADJUSTED TO 41.67 SCFM	
15:44:00	UNABLE TO MAINTAIN N_2 SUPPLY PRESSURE CONSTANT. PROBLEM WITH N_2 SUPPLY PRESSURE REGULATOR.	

SEPTEMBER 21, 1976 - DAY 265 - TEST 16001 → 05

16:40:00	N_2 REGULATOR NOW WORKING - N_2 FLOW ADJUSTED TO 41.67 SCFM	
18:30:30	TERMINATED 5 MIN SCANS	F/G
18:33:00	STARTED T-10 SCAN, PARA 3.2	
18:53:00	COMPLETED PARA 3.2.10	F/G

NOTE: TEST PARA 3.3 THRU 3.3.10 WILL NOT BE ACCOMPLISHED AT THIS TIME.

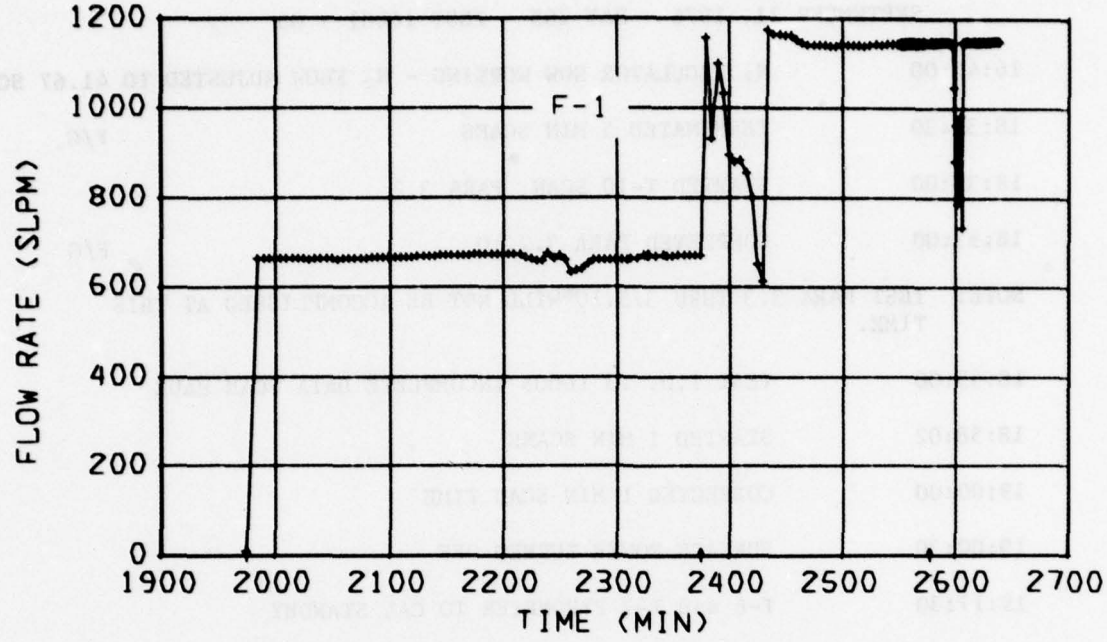
18:55:00	TEST I.D. TO 16005 INCOMPLETE DATA SCAN MADE	
18:58:02	STARTED 1 MIN SCANS	
19:00:00	CORRECTED 1 MIN SCAN TIME	
19:00:30	FURNACE POWER TURNED OFF	
19:17:30	T-6 and T-7 PYROMETER TO CAL STANDBY	
19:23:00	NOTICED N_2 FLOW FLUCTUATIONS	
19:30:00	N_2 FLOW APPEARS STABLE NOW	
20:00:30	DISCONTINUED 1 MIN SCANS N_2 FLOW AND SUPPLY TURNED OFF	F/G
20:15:00	STARTED 10 DATA SCANS	
20:18:00	END OF TEST AND DATA	F/G F/G

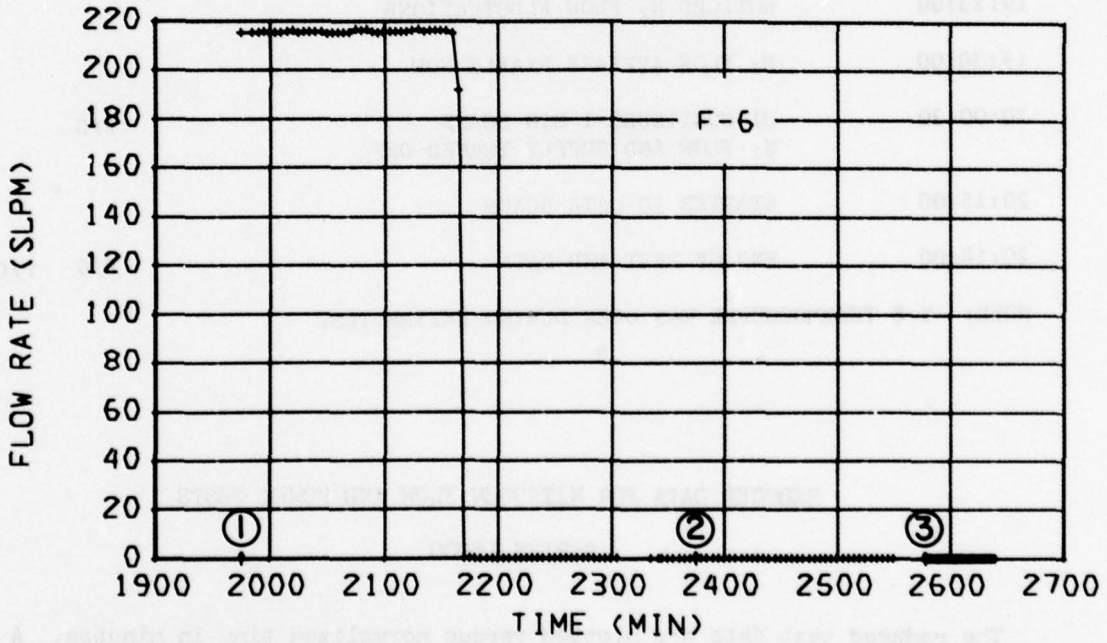
NOTE: T-8 THERMOCOUPLE WAS OPEN DURING ENTIRE TEST

REDUCED DATA FOR NITROGEN FLOW AND POWER TESTS

SERIES 16000

The reduced test data are plotted versus normalized time in minutes. A second reference scale at the bottom of each page gives the corresponding DAS or real time in hours. The label on each figure indentifies the parameter ploted (see Table B-1). The parameters are arranged in alphabetical and numerical order.





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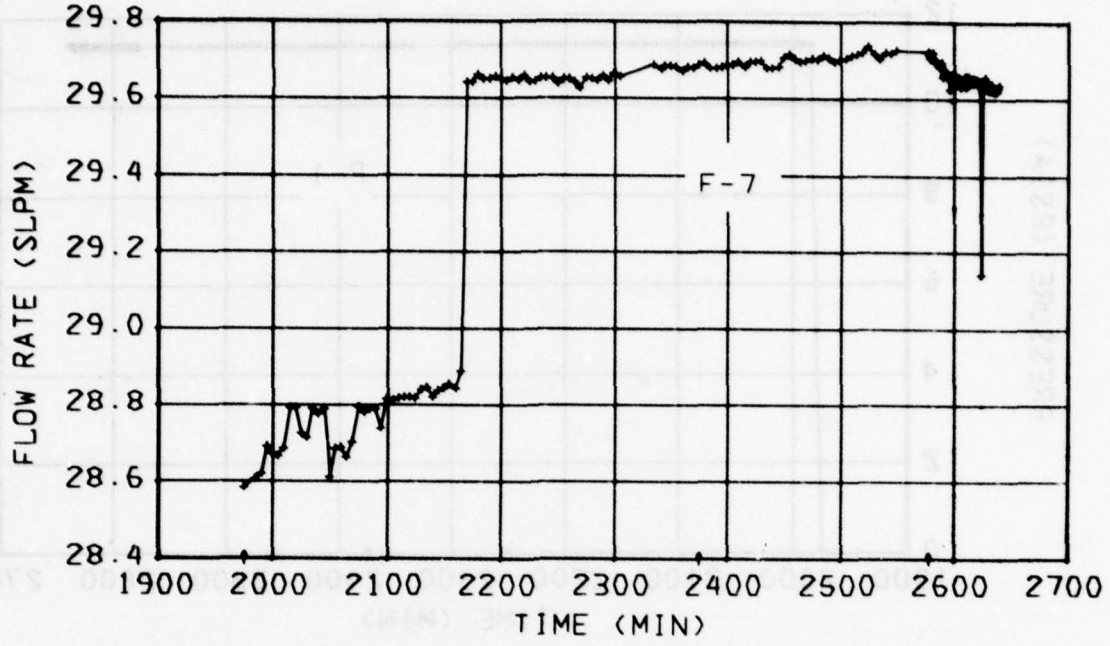

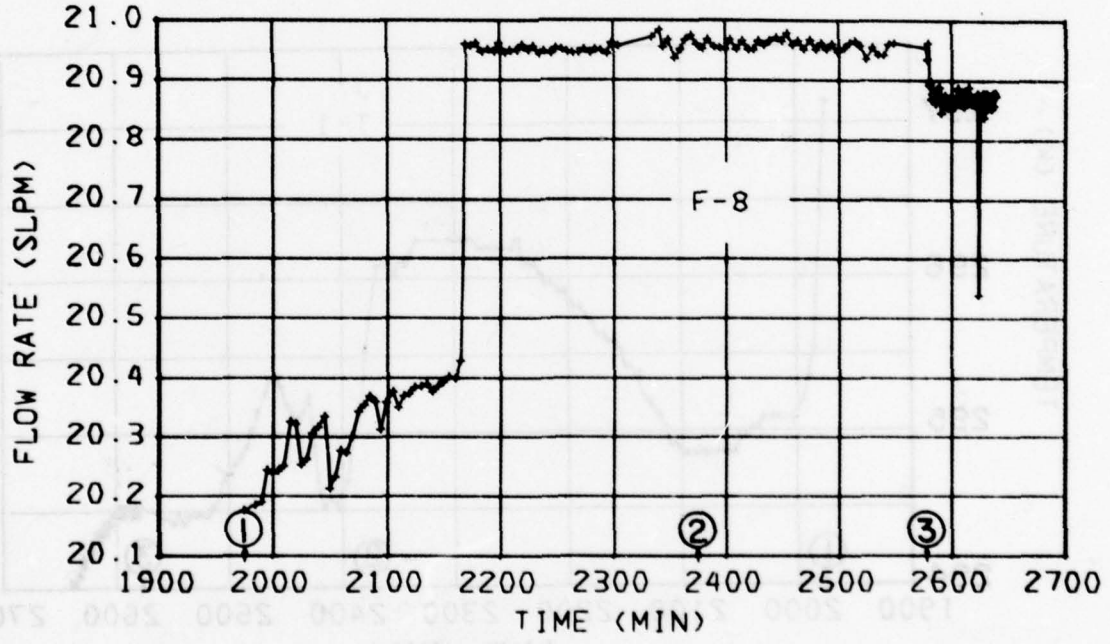

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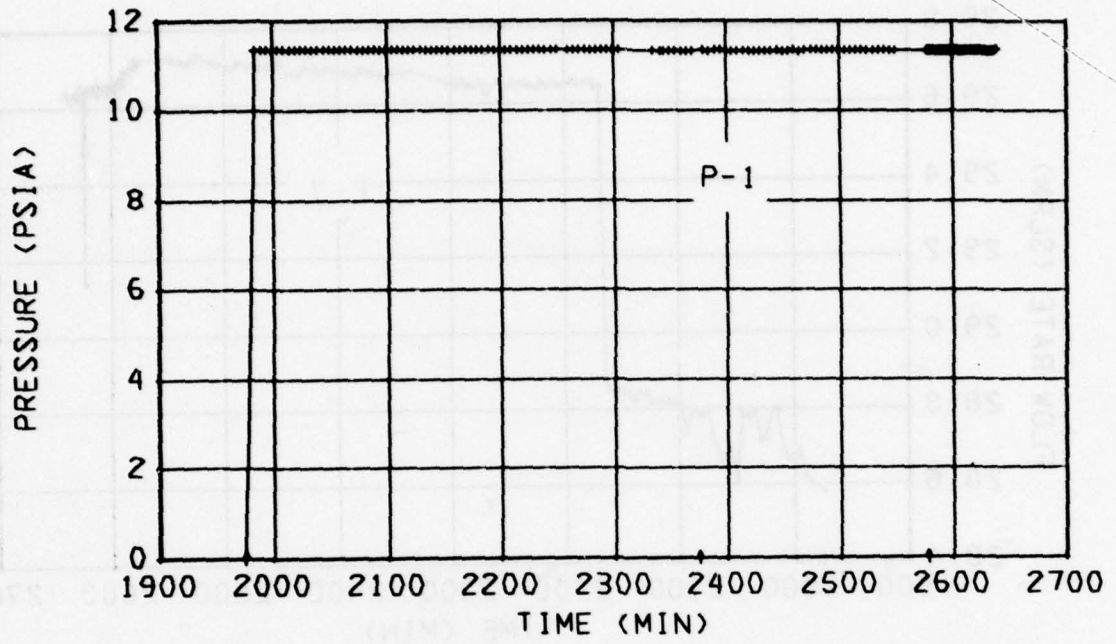

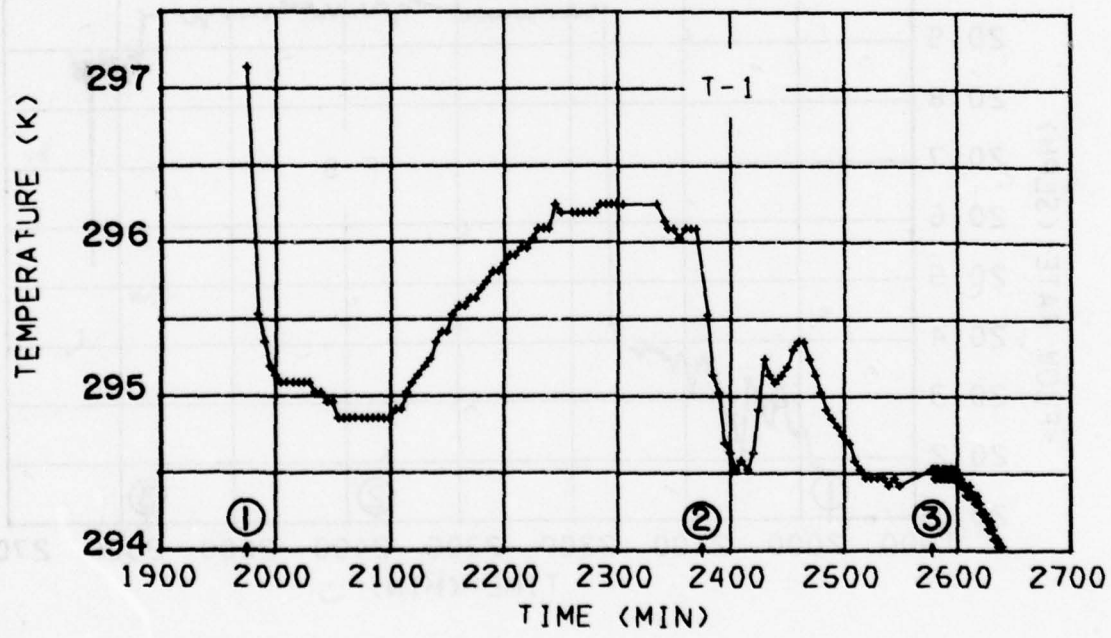

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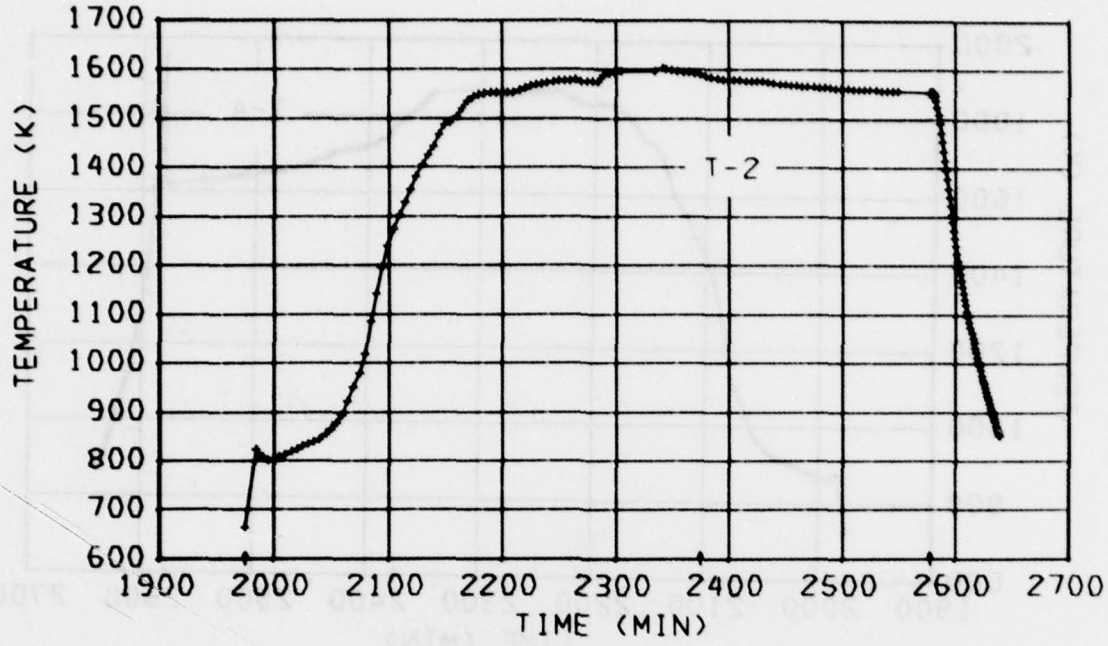

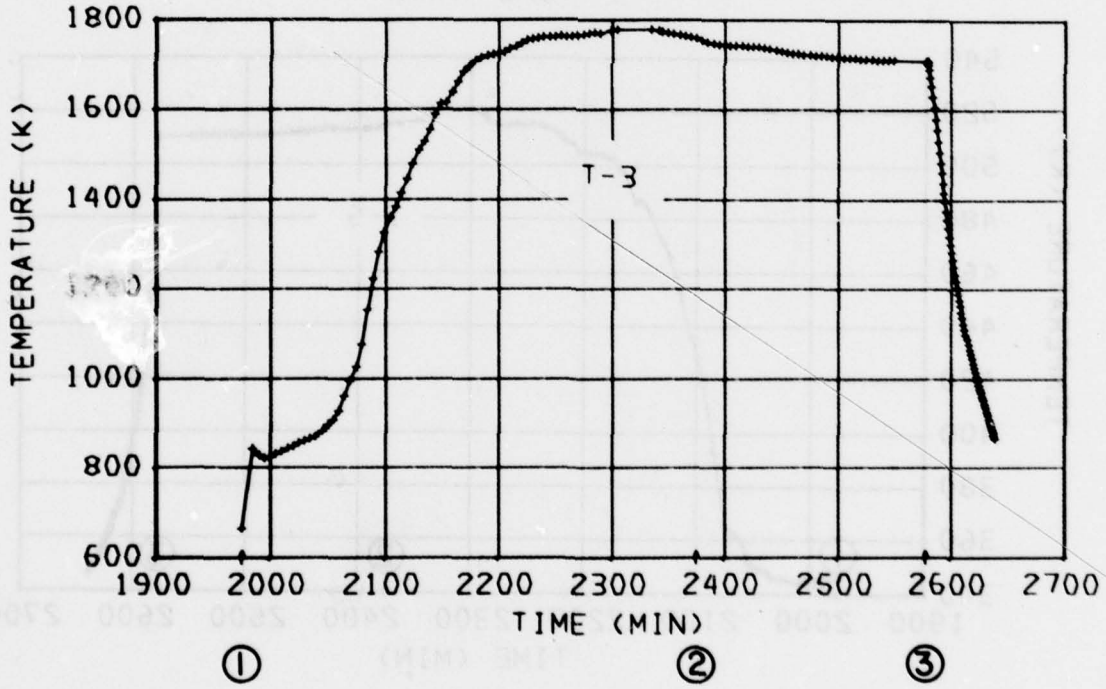

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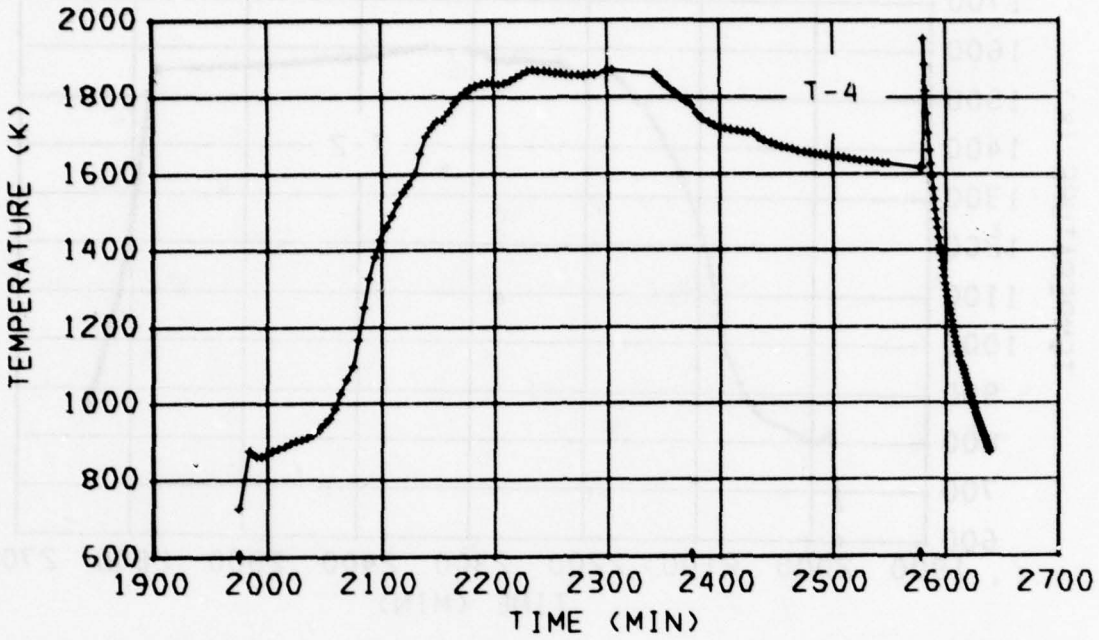

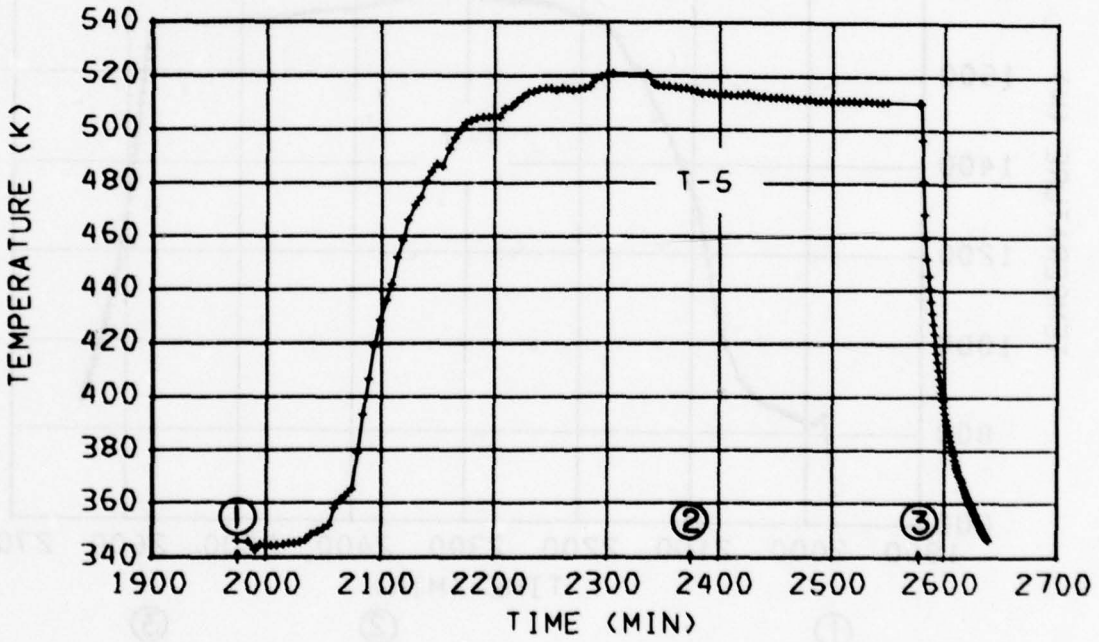

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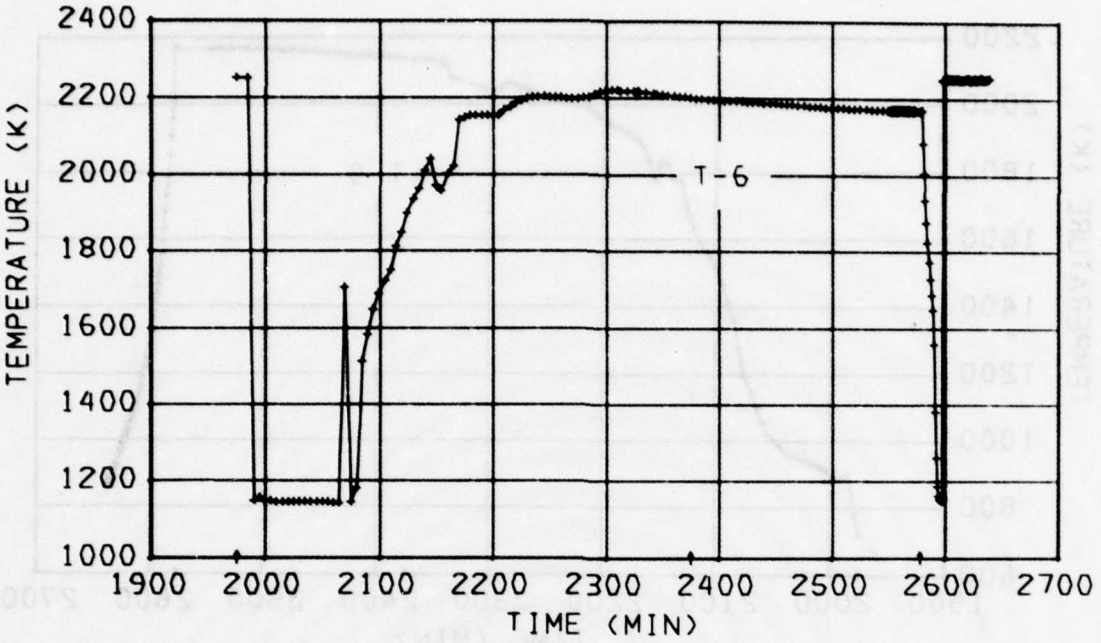

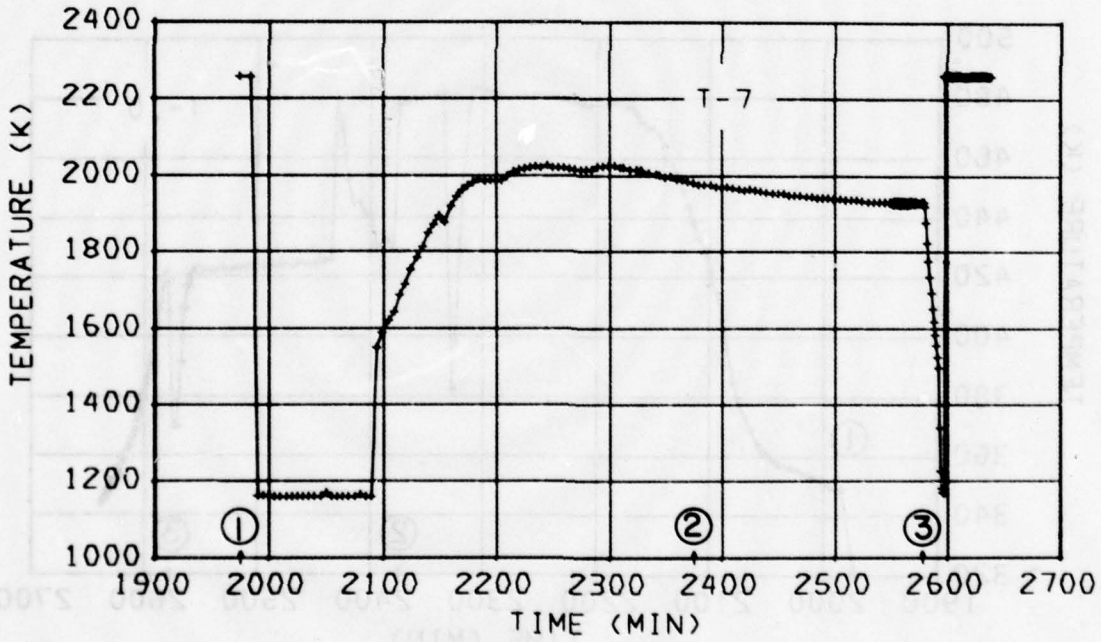

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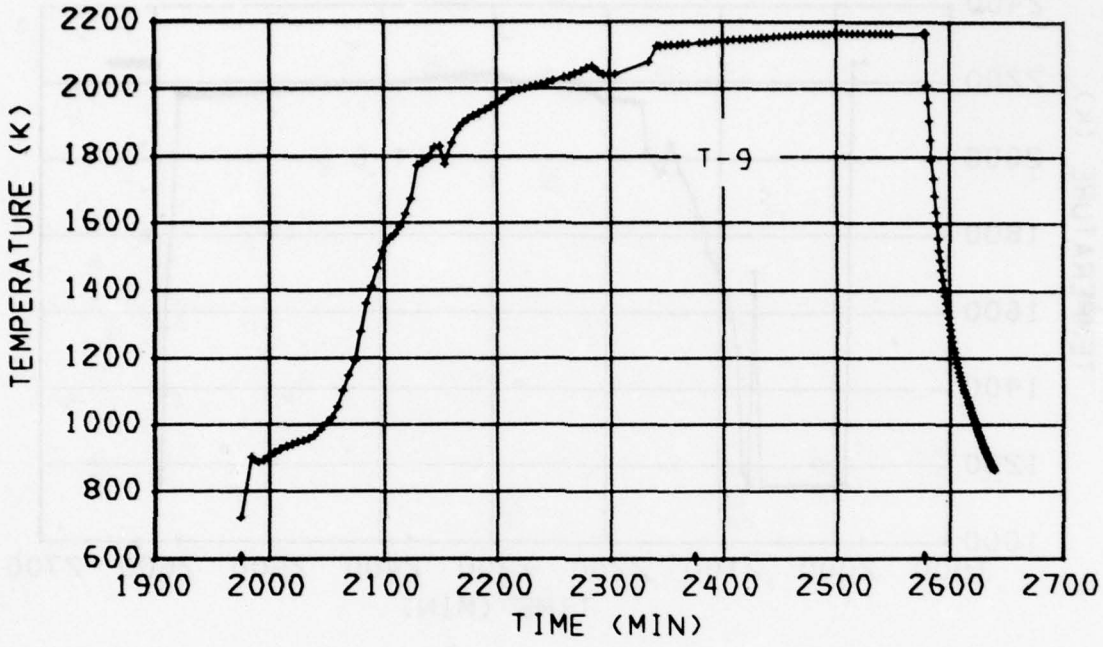

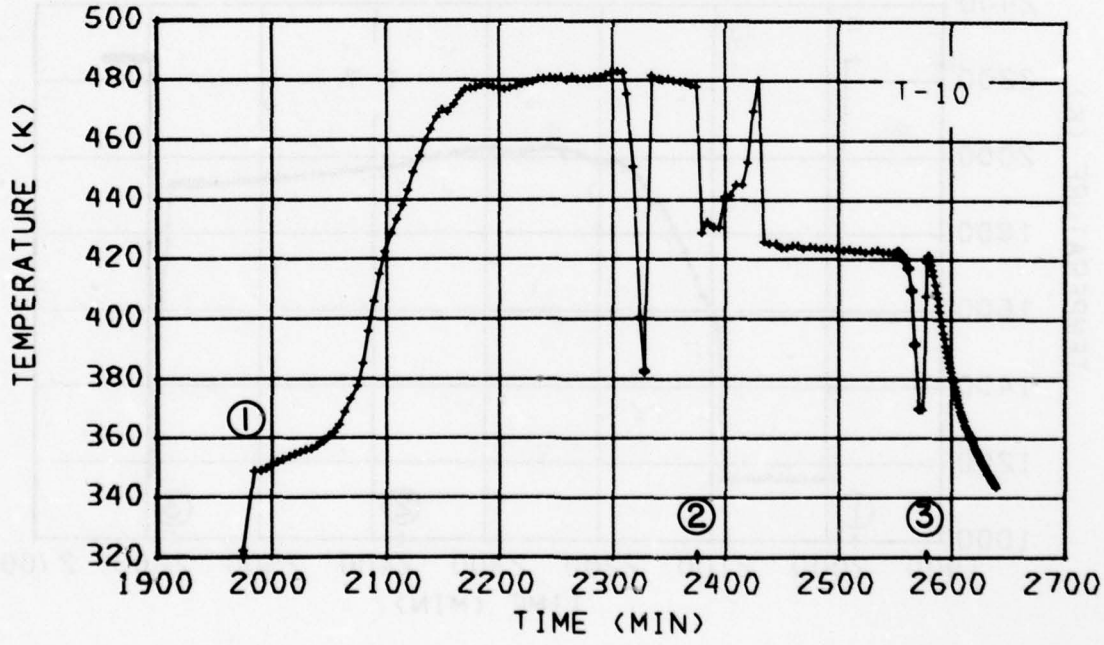

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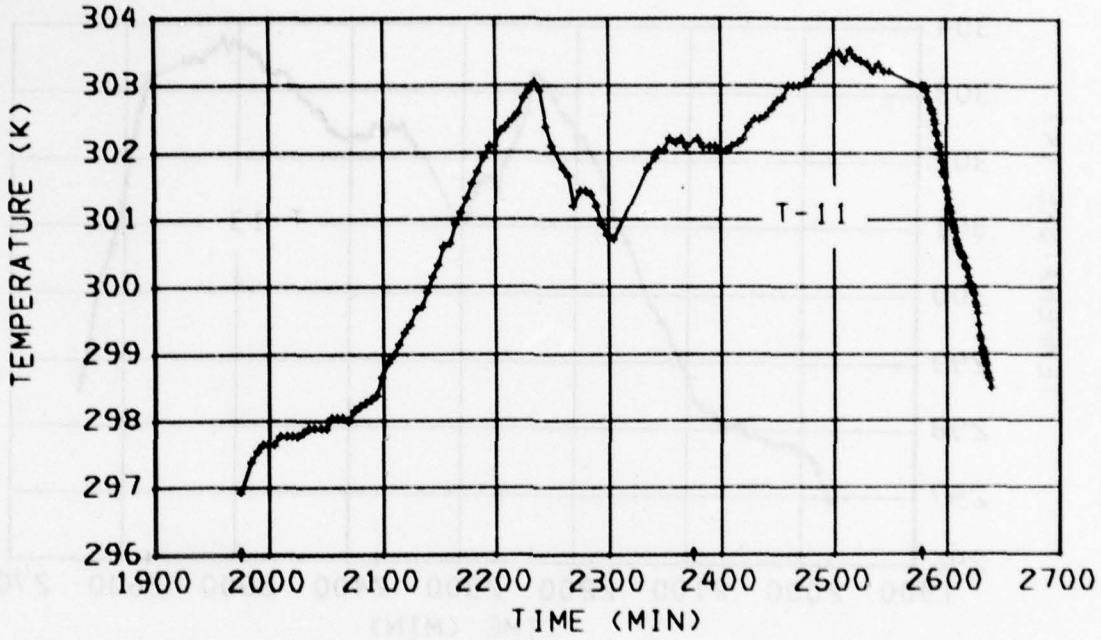

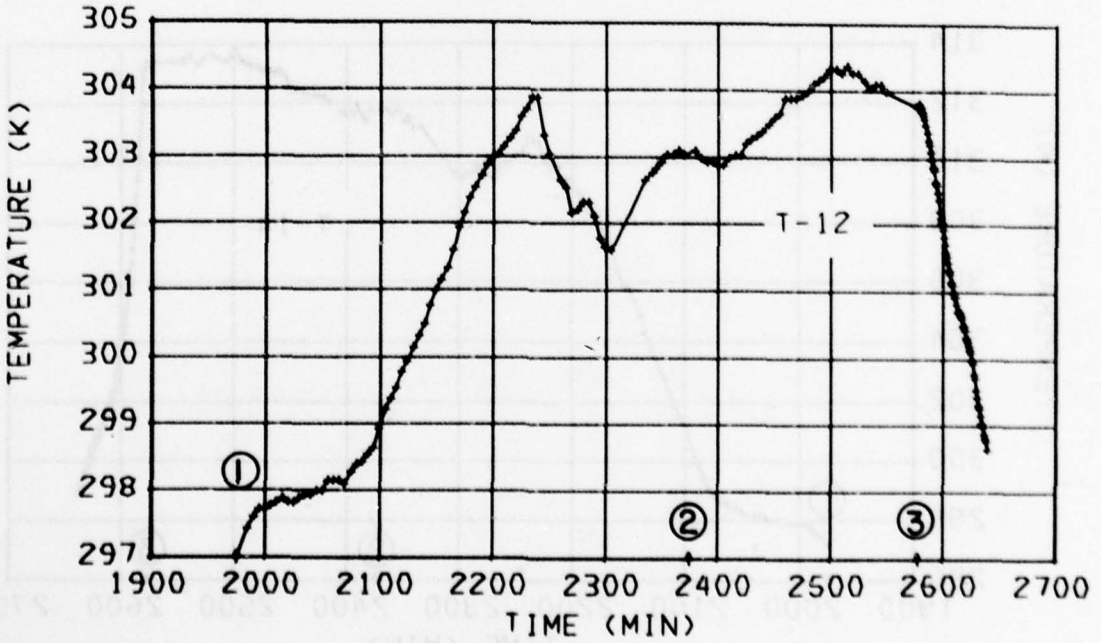

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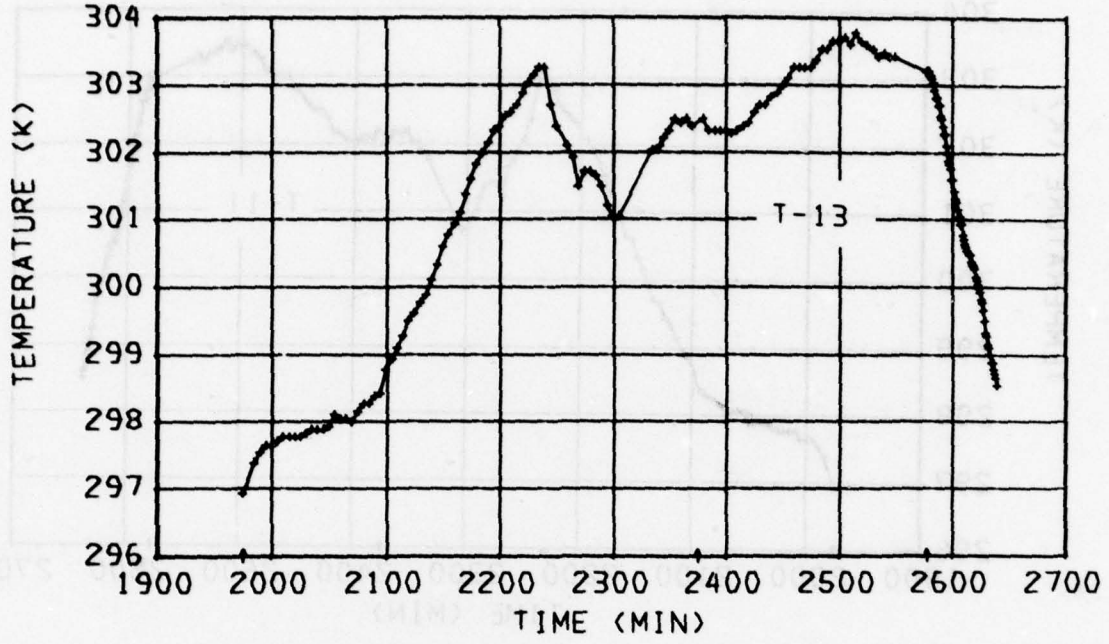

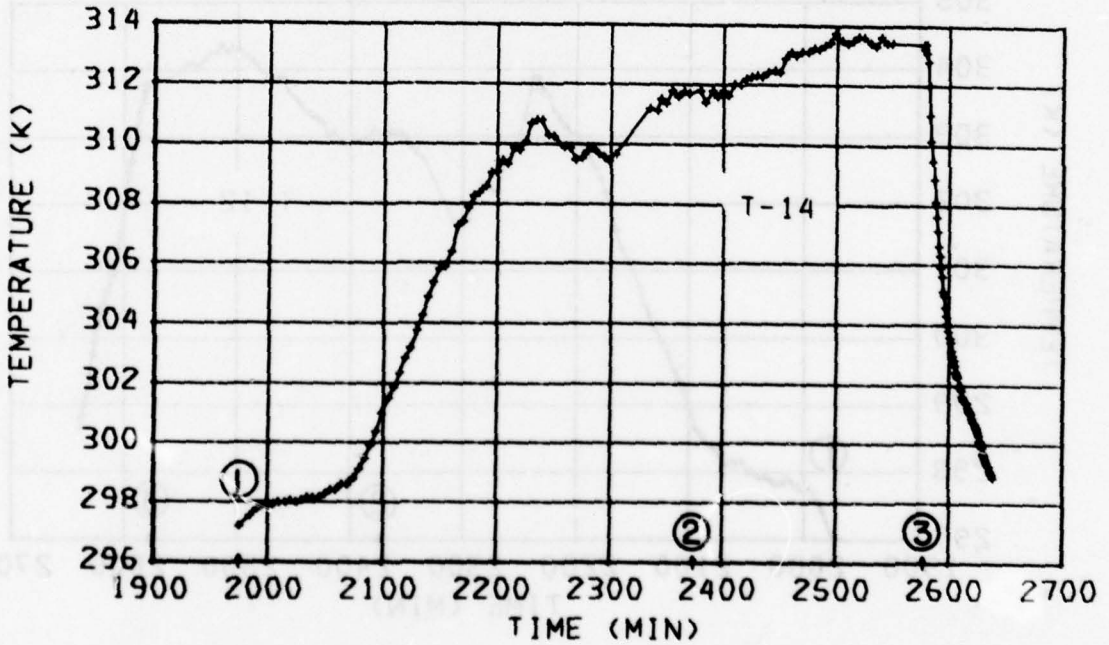

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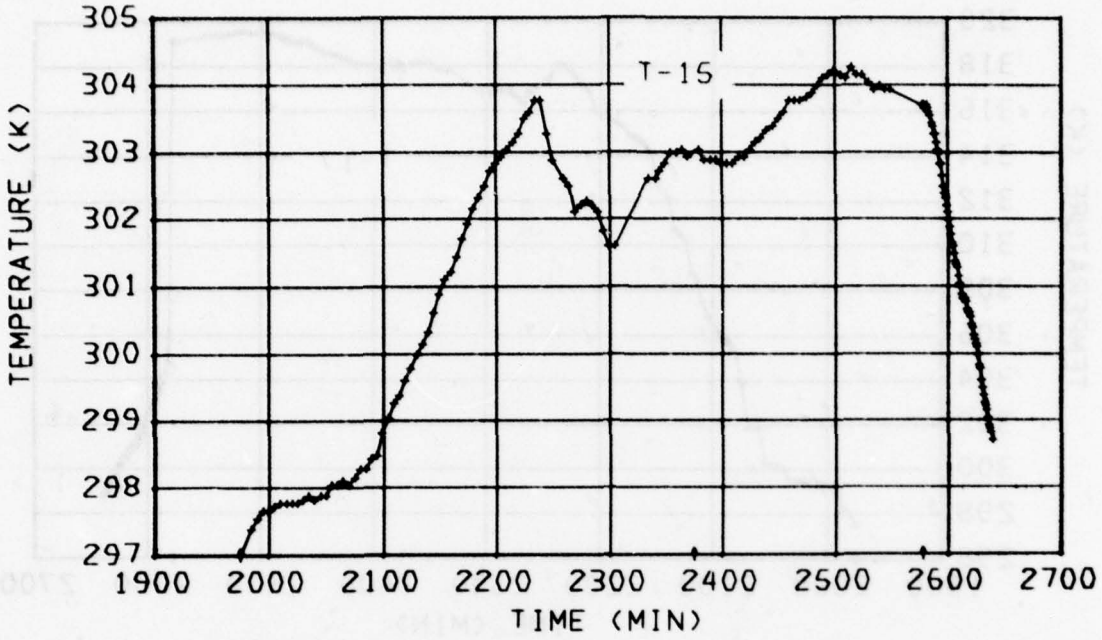

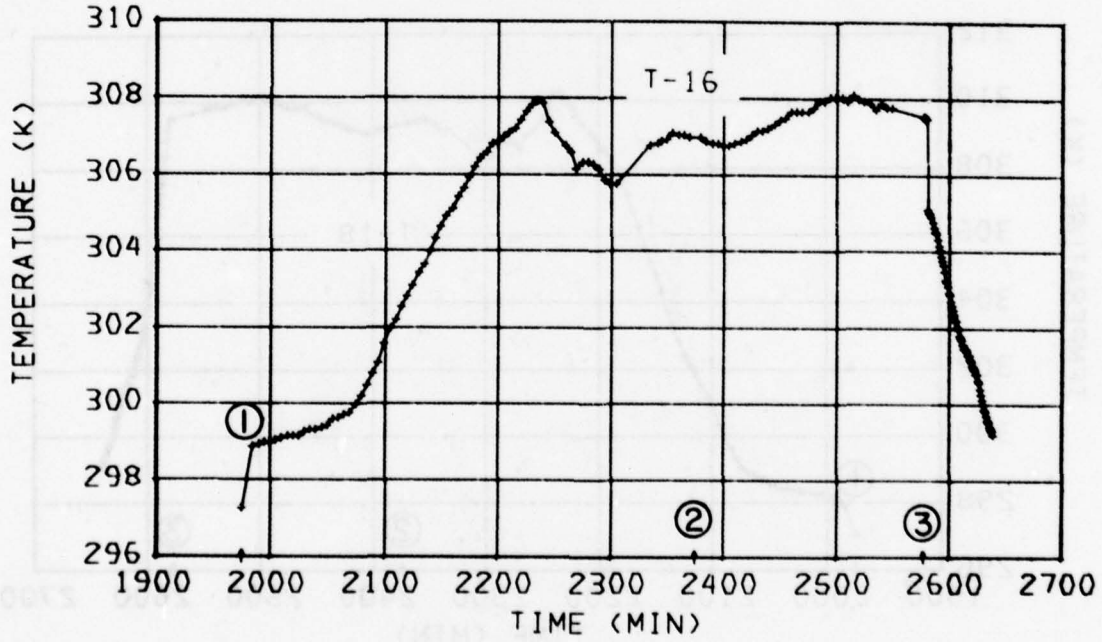

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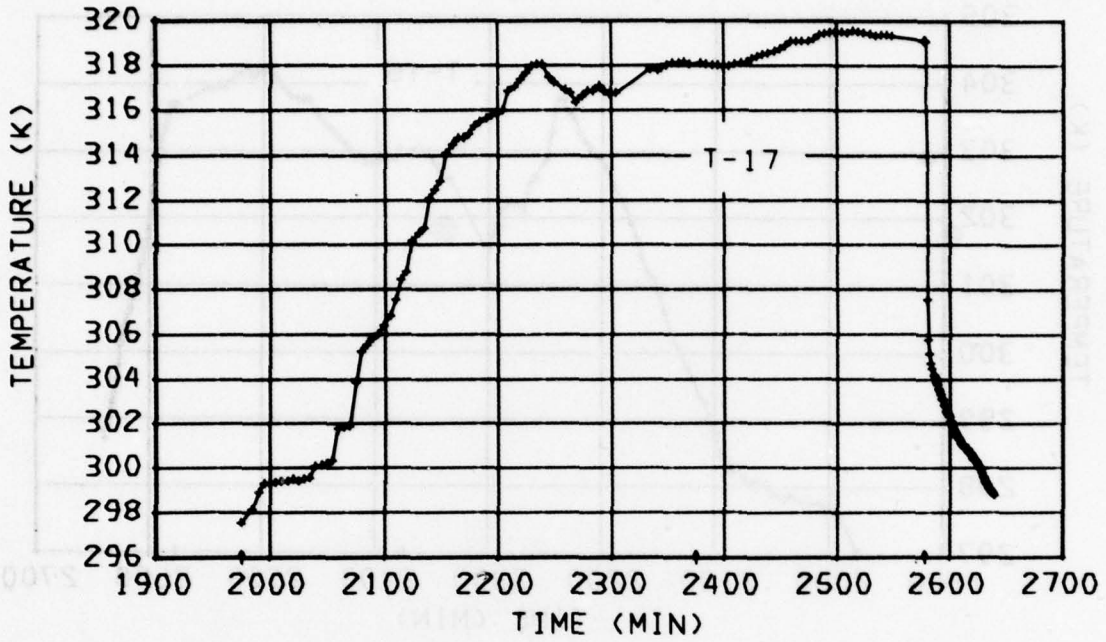

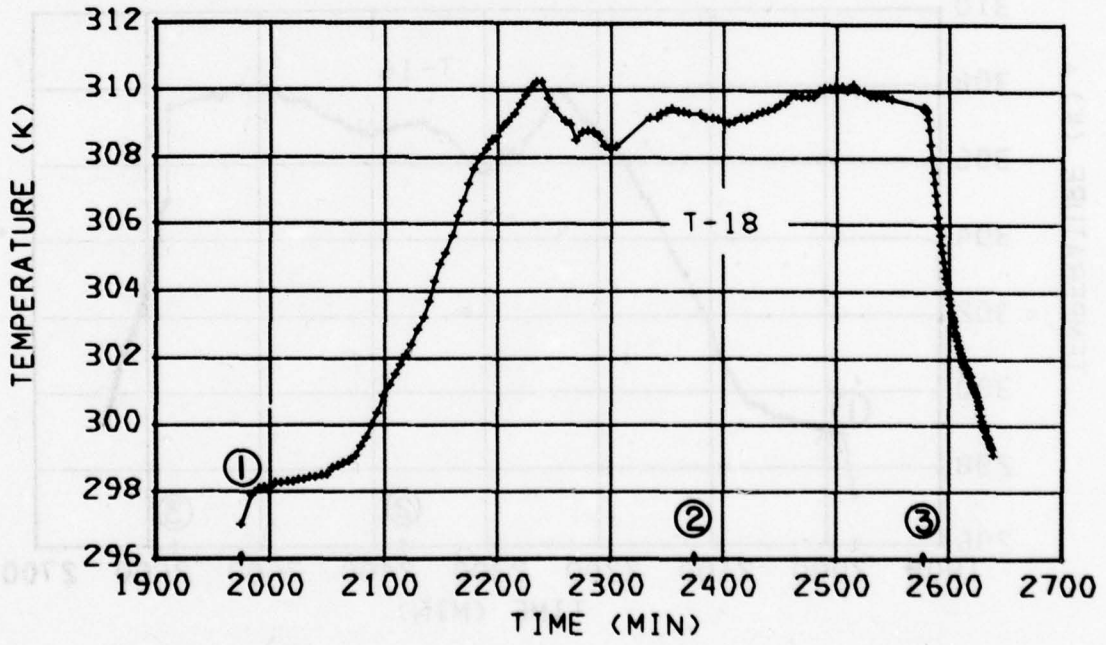

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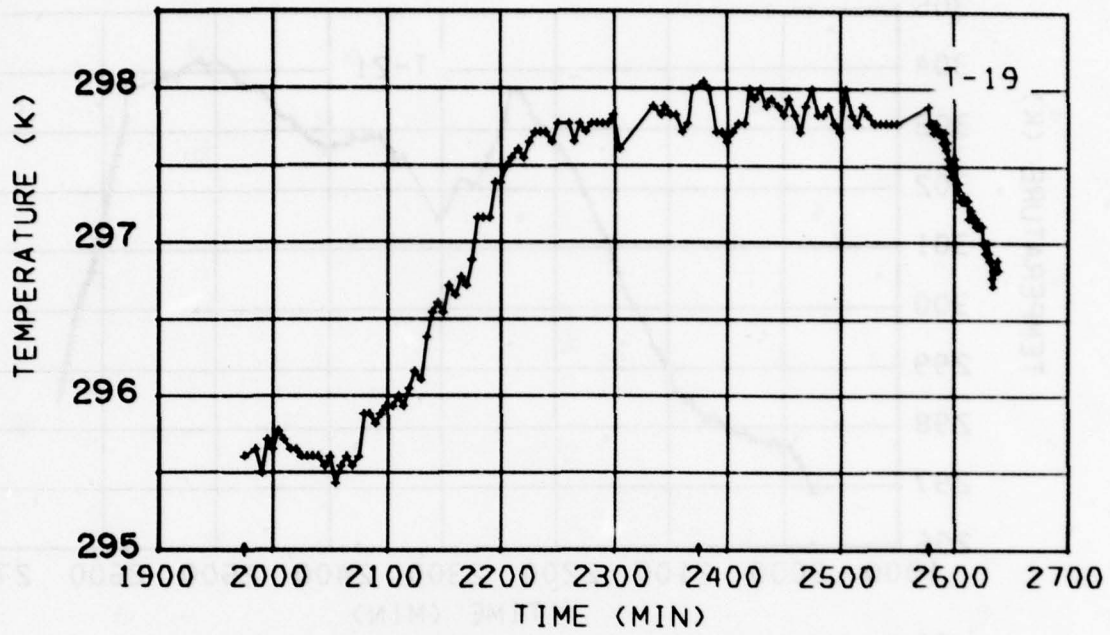

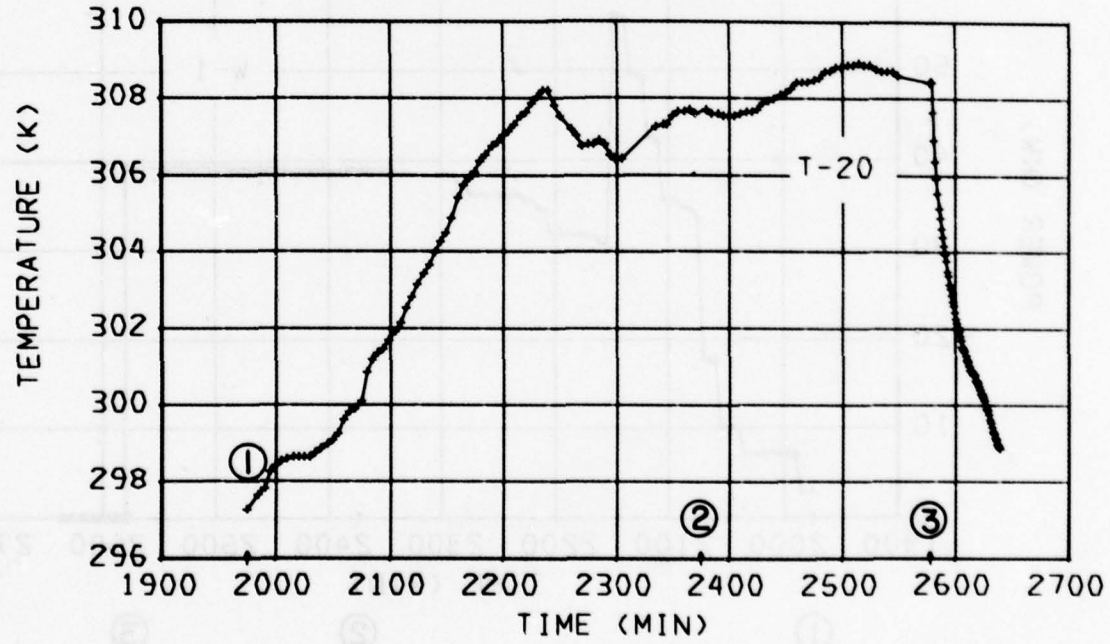

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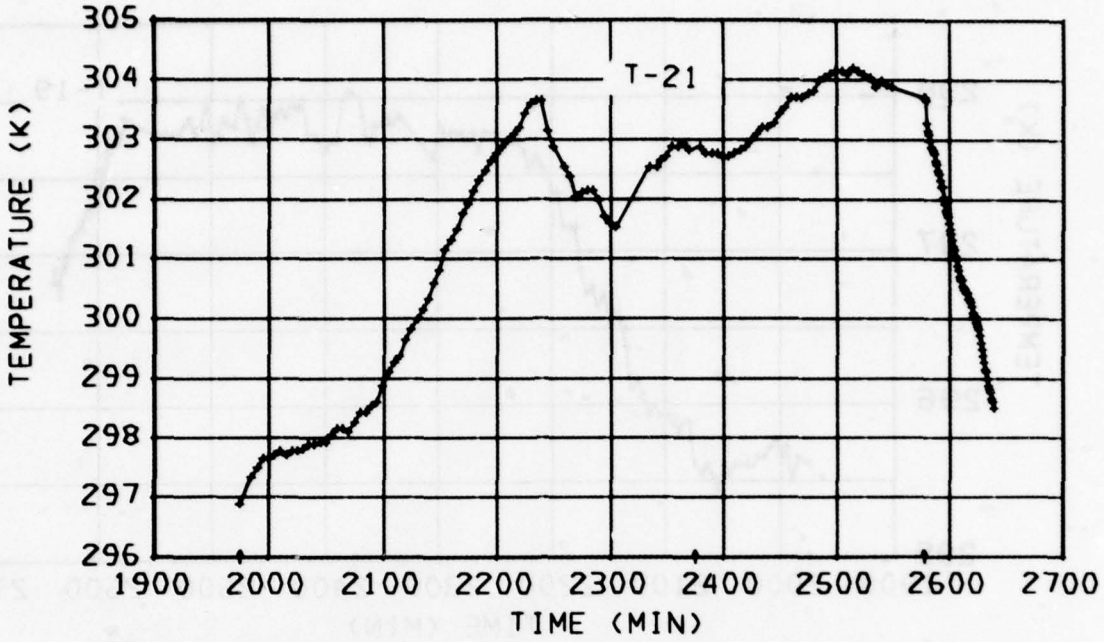
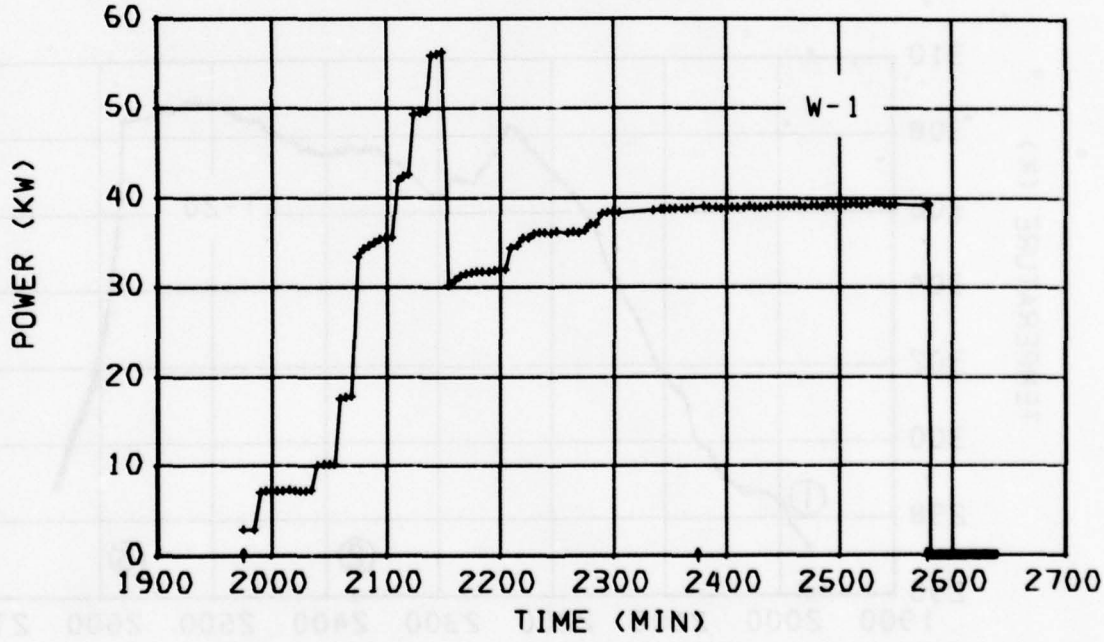
DAS TIME OR REAL TIME (H)

①,②,& ③ : SEE TEXT FOR TEST NO. DESCRIPTION





① ② ③

0740 0920 1100 1240 1420 1600 1740 1920 2100

DAS TIME OR REAL TIME (H)

①,②,& ③ : SEE TEXT FOR TEST NO. DESCRIPTION

the flow rate decreased from 1175 to 1138 ℓ/min. and stabilized at an average of approximately 1145 ℓ/min. for the duration of the test. The pressure regulator affected the flow rate again between 1919 and 1927 h. The decreased exhaust gas temperature during these periods verifies the decreased nitrogen flow rate. After the test, we found that the regulator was faulty and repaired it before the next series of tests.

The turbine flowmeter that measured the cooling water flow rate of the induction coil (F-6) failed at 1205 h. Its failure increased the pressure drop across it slightly and increased the flow rate through the other cooling water loops slightly. The data points (F-7 and F-8) at 1944 represent flow rate decreases of 0.5 and 0.3 ℓ/min. respectively.

The thermocouple that measured parameter T-4 became contaminated and started to give erroneous data after 1455 h.

The discontinuity in the T-6, T-7, and T-8 curves from 1150 through 1210 occured because the furnace power was turned off to adjust the reactive load of the furnace and consequently the power factor.

The first downward trend in parameter T-10 is shown by data taken during the exhaust temperature traverse at 1430 to 1448. At 1455 the thermocouple was again in its "parked" position. The data from 1535-1630 cover the period when the nitrogen flow rate was erratic because of the anamalous nitrogen pressure regulator. The exhaust temperature was traversed again at 1833-1852 h. These data are reflected in the negative trace during this time.

III. POWER AND COATING TESTS

The test identification numbers for these tests are in the 17000 series. The data presented herein are for tests 17002 through 17010.

B. Test Log - Power and coating tests

The test log for test series 17000 outlines pertinent events and their time of occurrence. It is enclosed so that the reader may correlate these events with the data graphs. The original test log is on file at LASL.

C. Test Data - Power and coating tests

The following figures cover that part of the test in which power was applied to the furnace. It was not convenient to present the data taken during other parts of the test in this format, so parts of the data appear in Sec. III of this report. All data were recorded on magnetic tape, which is on file at LASL.

17000 THRU 17010 TEST LOG

TEST 17000 THRU 17010
DAY 287 - October 13, 1976

TIME	REMARKS	F/G
154500	Ten scans per paragraph 1.10 with power off	
154730	Power adjusted to 20 kW on control meter	
154800	1 Scan	
154900	1 File gap per paragraph 1.10	F/G
161300	1 File Gap - Mistake	F/G
161500	Set test I.D. to 17002 Test procedure 2.0 thru 2.6.39 not done at this time	
161830	T-6 and T-7 pyrometers switched to run	
162500	5 min scans initiated per paragraph 3.1.2	
162600	1 extra scan made	
16300	Back to 5 min scans	
163500	Incomplete scan made - checking N_2 flow	
163600	1 full scan made	
164000	1 full scan made Have faulty indications on N_2 flow meter - trouble-shooting system (F-1)	
181700	N_2 flow now appears to have settled down system back on (F-1)	
182000	1 min scans initiated	
183230	N_2 flow fluctuating again. Turned meter off	
184230	Turned N_2 system on again (F-1)	
192400	Turned N_2 system off - fluctuating (F-1)	
194300	Added 5.3 uf to capacitor have 10.23° phase angle indicated by power measurements	

195000	Made resistance checks on N_2 transducer, checks are bad per operating manual. Turned system back on.	
203600	Turned N_2 system off - erratic - (F-1). System seems bad after long warmup	
204700	Discontinued 1 min scans	
204800	1 file gap per paragraph 3.1.7 set test I.D. to 17003	F/G
205000	Initiated 1 min scans	
205500	N_2 flow system turned on (F-1)	
215700	N_2 flow system turned off, fluctuating (F-1)	
221330	N_2 flow system turned on (F-1)	
223100	Discontinued DAS scan per paragraph 3.2.8 1 file gap	F/G
223300	Set test I.D. to 17004	
223400	Added 2.6 uf to capacitor (power off)	
223500	Removed 1.3 uf from capacitor (power off)	
223800	Set power to 72 kW using DAS voltage	
224200	Set N_2 flow rate to 30 SCFM	
224500	Initiated 5 min scans per paragraph 3.3	
225000	Noticed what appears to be gap in felt in area of T-3	
230300	N_2 flow system turned off (F-1)	
231300	Changed T-6 from low to high range, file gap	F/G
232500	N_2 flow system turned on (F-1)	
233100	Increased power 3 kW on control meter	
234400	Increased power 3 kW on control meter	

TEST 17000 THRU 17010
DAY 288 - October 14, 1976

TIME	REMARKS	F/G
002300	Turned N_2 flow system off - erratic (F-1)	
004500	Discontinued 5 min scans per paragraph 3.3.9 1 file gap Turned N_2 flow system on (F-1) Test I.D. To 17005	F/G
005000	Initiated 5 min scan per 4.1.3	
005400	Started CH_4 flow	
010000	Started MTS flow	
010800	Reduced power 2 kW on control meter	
13624	Turned N_2 flow system off (F-1)	
015520	Turned N_2 flow system on (F-1)	
015600	Increased power 1 kW on control meter	
021000	Increased power 1 kW on control meter	
021200	Readjusted T-6 pyrometer head temperature increased 40°C	
022400	Turned N_2 flow system off (F-1)	
022700	Increased power 1 kW on control meter	
023800	Turned N_2 flow system on (F-1)	
031600	Increased power 1 kW on control meter	
032700	Turned N_2 flow system off (F-1)	
035100	Increased power 1 kW on control meter	
040100	Turned F-1 on	
042300	Turned F-1 off	
042800	Turned F-1 on	

043000	Discontinued 5 min scan per paragraph 4.1.9 1 file gap Test I.D. to 17006	F/G
043200	Initiated 5 min scan per paragraph 4.2.4	
043247	SV-1 to off	
043300	SV-3 to off	
043400	Discontinued scan to set N_2 flow per paragraph 4.2.7	
045500	Power to furnace off - added 2.7 uf capacitor	
050000	Initiated 5 min scan	
050200	Furnace power turned on	
050525	Changed T-6 from high to low range 1 file gap	F/G
050920	Turned F-1 off	
051425	Changed T-6 from low to high range 1 file gap	F/G
054100	Increased power 3 kW on control meter	
055800	Increased power 3 kW on control meter	
061900	Increased power 3 kW on control meter	
063245	Added 1.3 uf capacitor (power off)	
063600	Power turned on	
070152	Power off added .64 uf capacitor	
070230	Sparks from capacitor bus bar Tightened all capacitor nuts-removed .64 uf	
070800	Initiated 5 min scan - 1 scan only	
071500	Power turned on	
072500	Initiated 5 min scan	
072900	Turned F-1 on	
074000	Increased power 2 kW on control meter	

17000 THRU 17010 TEST LOG (cont'd)

075100	Increased power 3 kW on control meter	
083300	Discontinued scan per paragraph 4.2.11 1 file gap Test I.D. to 17007	
083500	Initiated 5 min scan per paragraph 4.3.4	F/G
083800	Started CH_4 flow per paragraph 4.3.5	
084800	Started MTS flow per paragraph 4.3.7 Ratio over range at this time	
90030	Decreased power 2 kW on control meter	
090200	Ratio now on scale	
091600	Decreased power s kW on control meter	
093100	Turned F-1 off	
101600	Turned F-1 on	
101700	Increased power 2 kW on control meter	
111200	Increased power 2 kW on control meter	
114900	Changed CH_4 supply bottle	
115100	Turned F-1 off	
120700	Turned F-1 on	
122300	Removed room temp T/C to measure bell jar flange temperature in area above vacuum pump flange	
124600	Discontinued scan per paragraph 4.3.9 1 file gap Test I.D. to 17008	
125000	Initiated 5 min scans - 1 scan	F/G
125300 thru 1420	Tried several combinations of capacitors to obtain power factor of 1	
142314	Resumed 5 min scans	

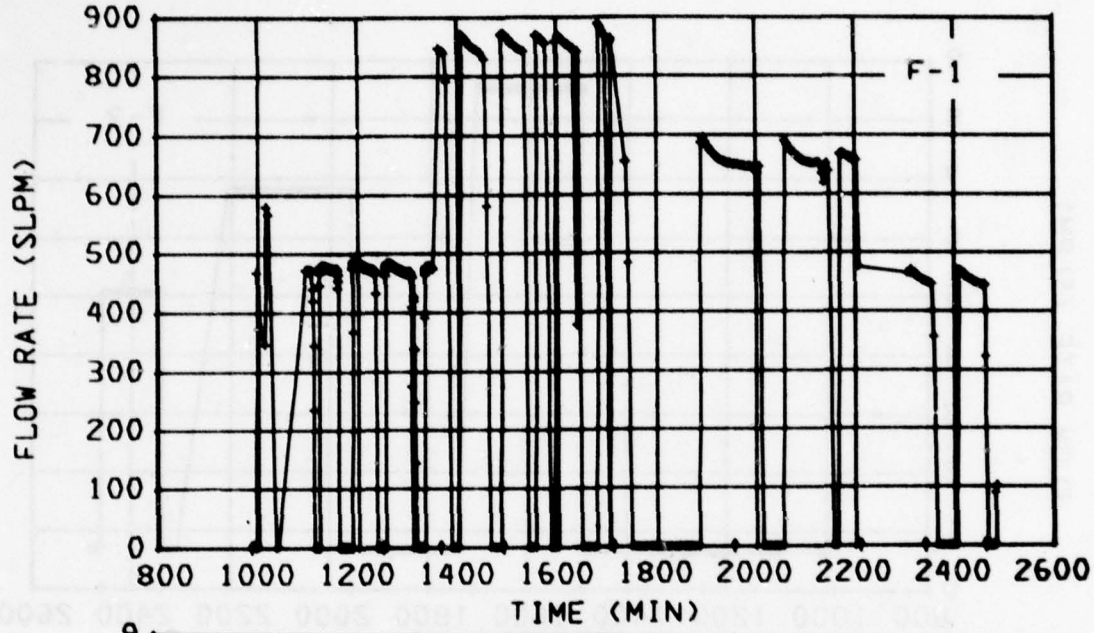
145300	Stabilized temperature per paragraph 4.4.19 Discontinued 5 min scan Test I.D. to 17009 1 file gap	F/G
145500	Initiated 5 min scan per paragraph 4.5.4	
145540	CH_4 Flow on	
150540	MTS flow on Ratio over range (30.2%)	
152030	Reduced power 1 kW by control meter	
152100	Turned F-1 off	
160600	Turned F-1 on	
160900	Power phase angle approximately 25.76°	
161000	T-7 indicates 1746°C	
161530	Power increased 1 kW by power meter power limiting at this setting	
163000	Ratio now 30.4% over range	
170100	Discontinued 5 min scans per paragraph 4.5.9 1 file gap Test I.D. to 17010	F/G
170201	Initiated 1 min scans per paragraph 5.4	
170252	MTS flow off	
170256	CH_4 flow off	
170330	High frequency power off	
170500	Turned F-1 off	
170620	F-1 on DAS to single point	
170900	N_2 flow set to 3.3 SCFM	
171230	F-1 turned off T-6 turned to low range 1 file gap	F/G

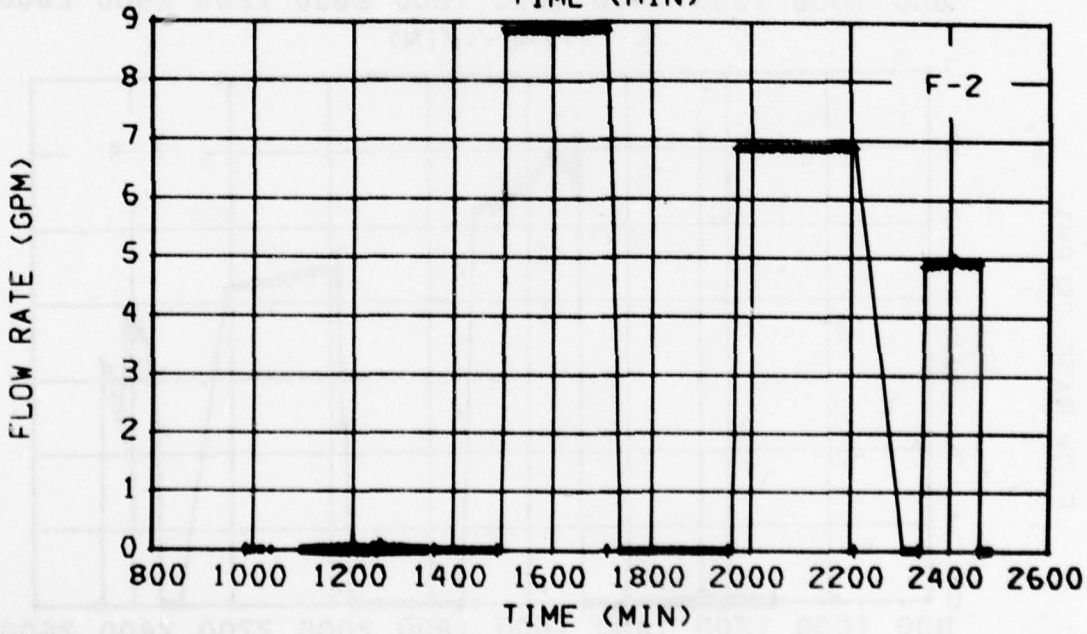
171400	T-6 low intensity light	
171500	Opened T-6 aperature - full	
172300	Turned F-1 on	
172500	T-6 and T-7 low intensity lights on	
173000	DAS recording F-1 as .2601 V should be .198 V	
180930	DAS scan off	
	1 file gap	F/G
181000	Set both pyrometers to "cal" mode	
181500	Purging MTS lines with H_e Purging CH_4 lines with N_2	
181700	10 continuous scans at zero condition	
	1 file gap	F/G
	1 file gap	F/G
	Total - 17 file gaps on data tape	

REDUCED DATA FOR POWER AND FIRST COATING TEST

SERIES 17000

The reduced test data are plotted versus normalized time in minutes. A second reference scale at the bottom of each page gives the corresponding DAS or real time in hours. The label on each figure identifies the parameter plotted (see Table B-1). The parameters are arranged in alphabetical and numerical order.





① ②③④ ⑤ ⑥ ⑦⑧⑨

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DAS TIME OR REAL TIME (H)

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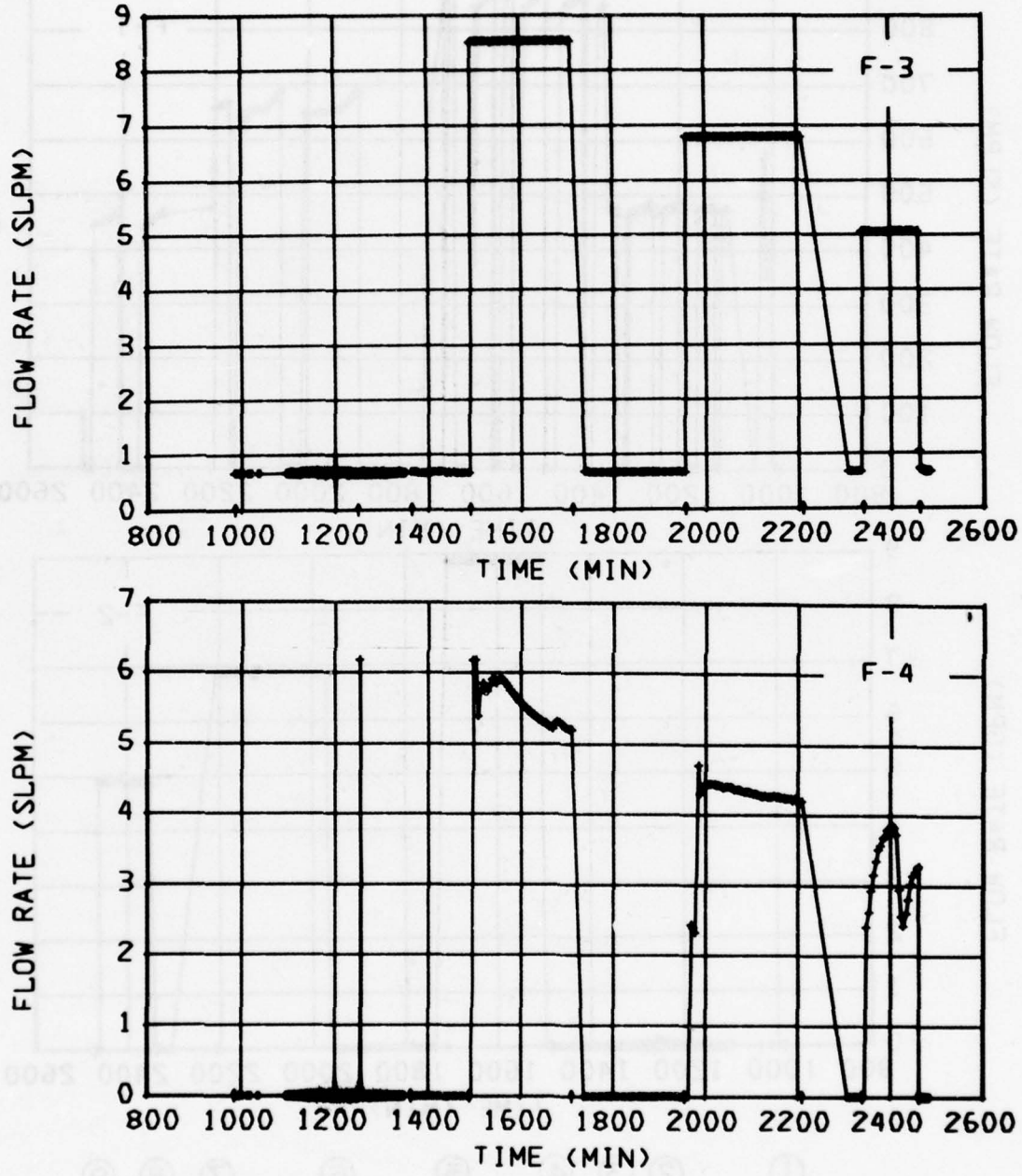

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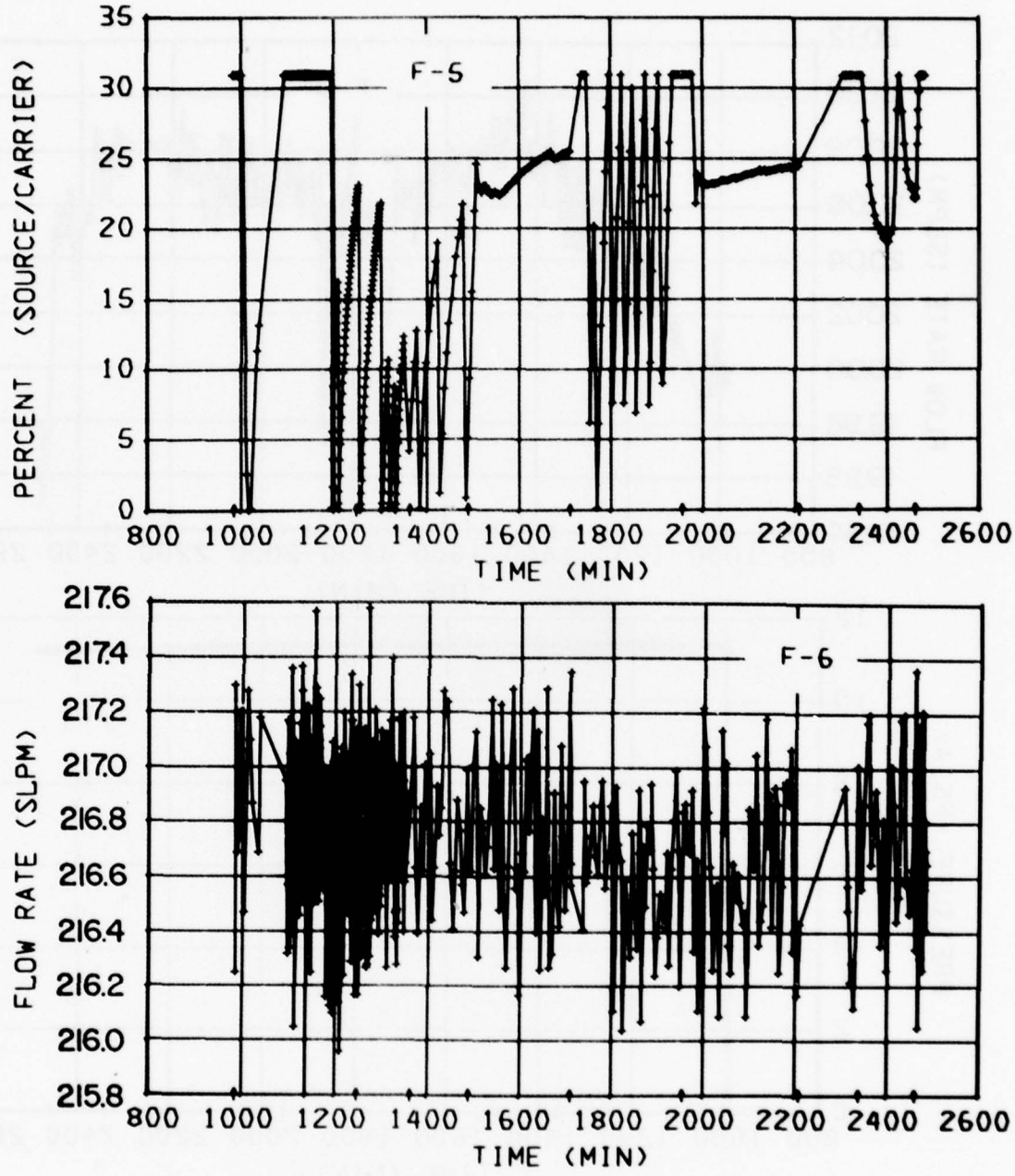

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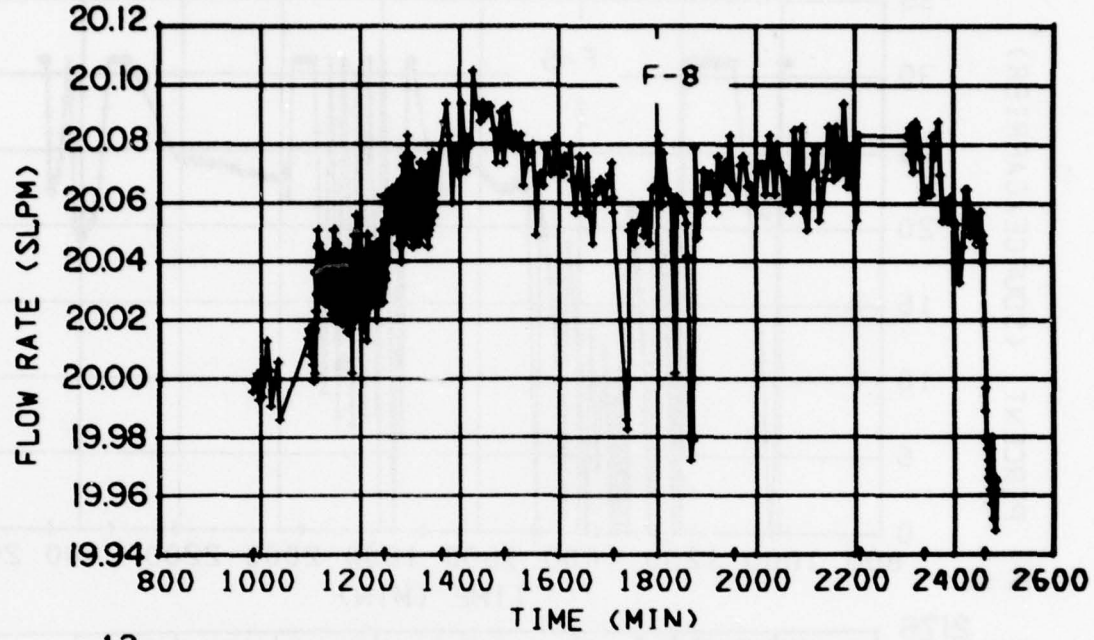

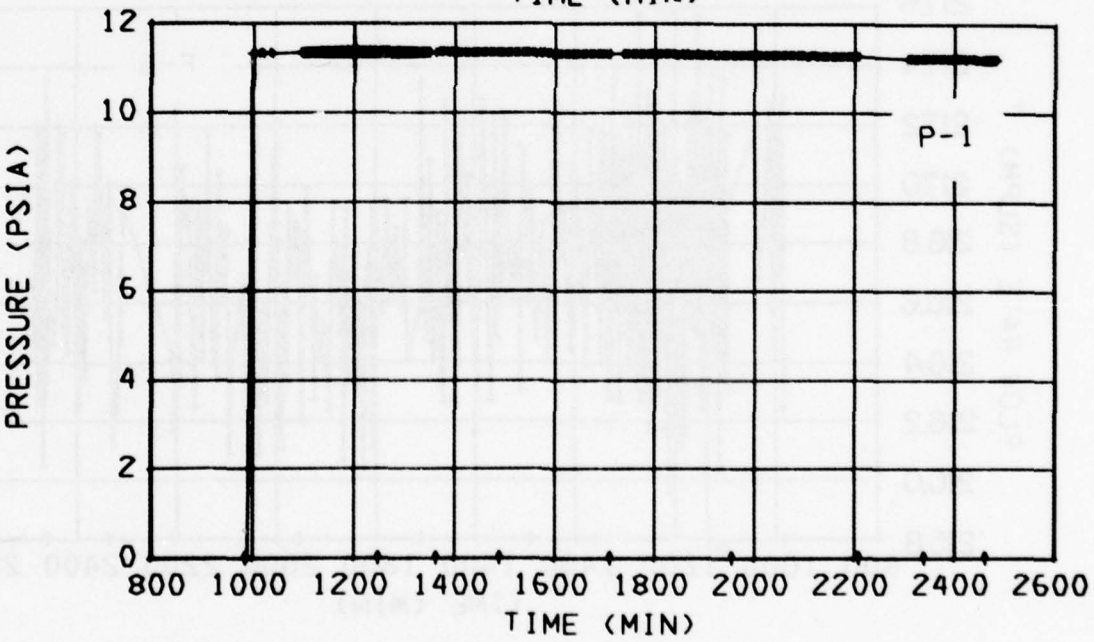

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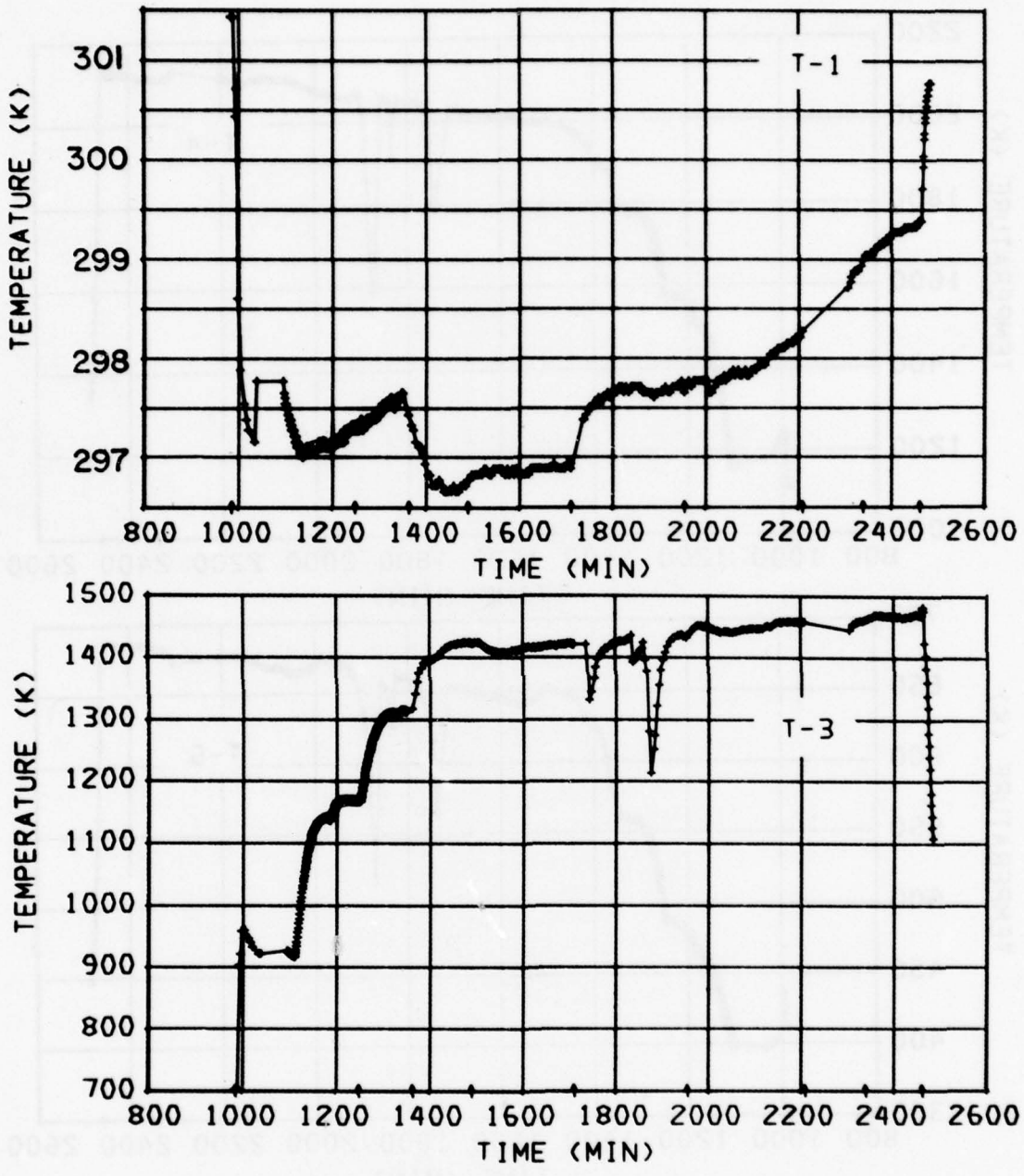

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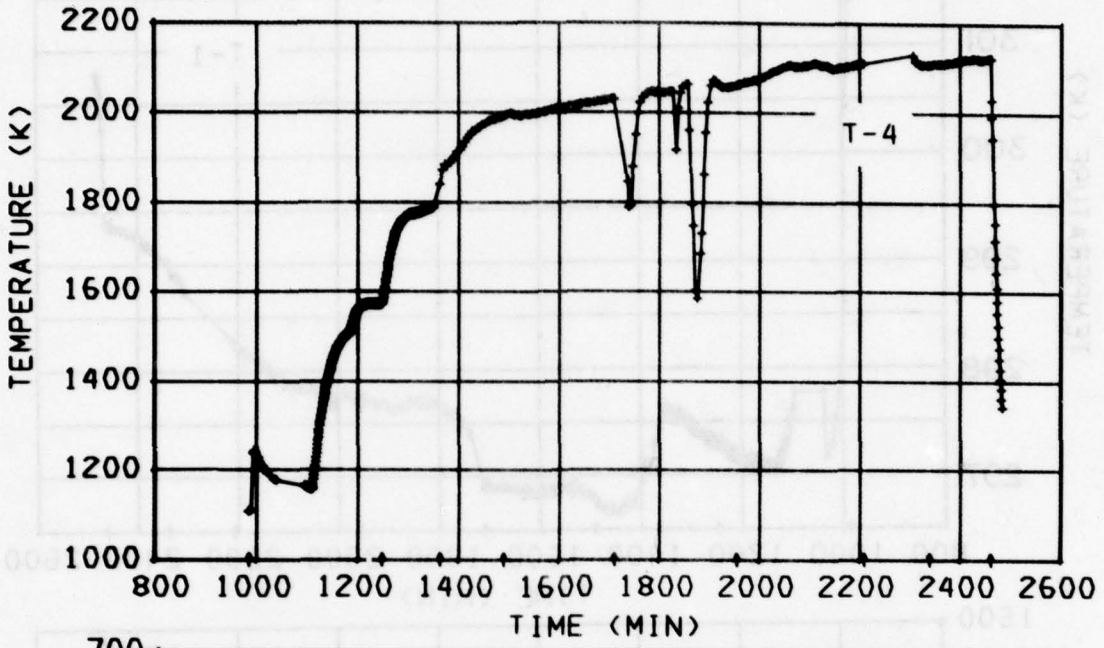

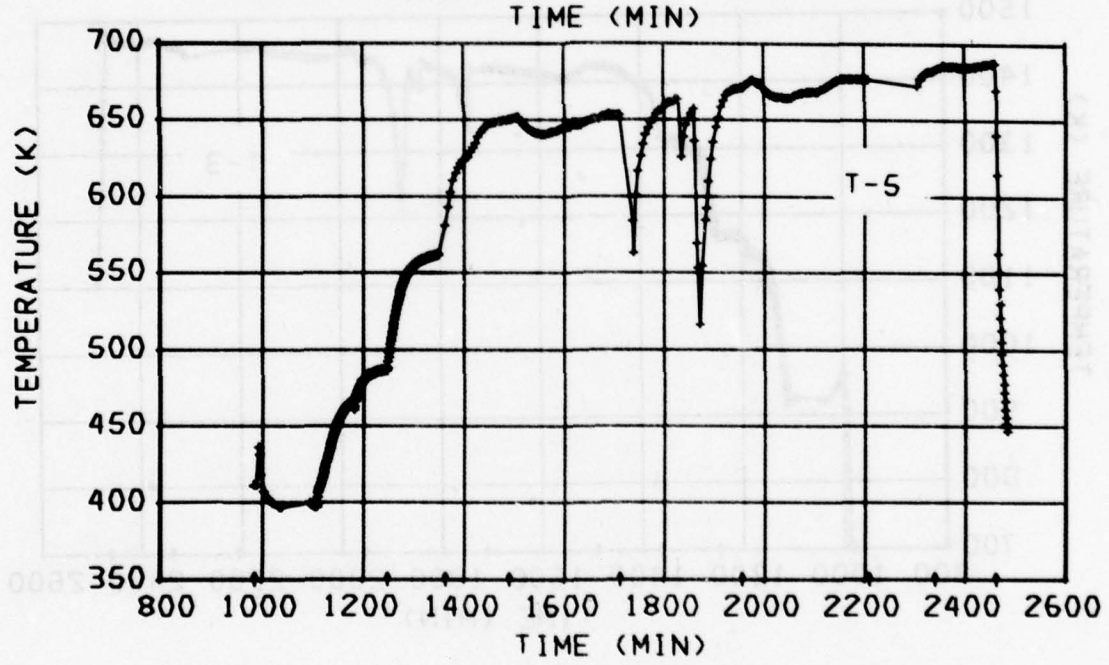

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DAS TIME OR REAL TIME (H)

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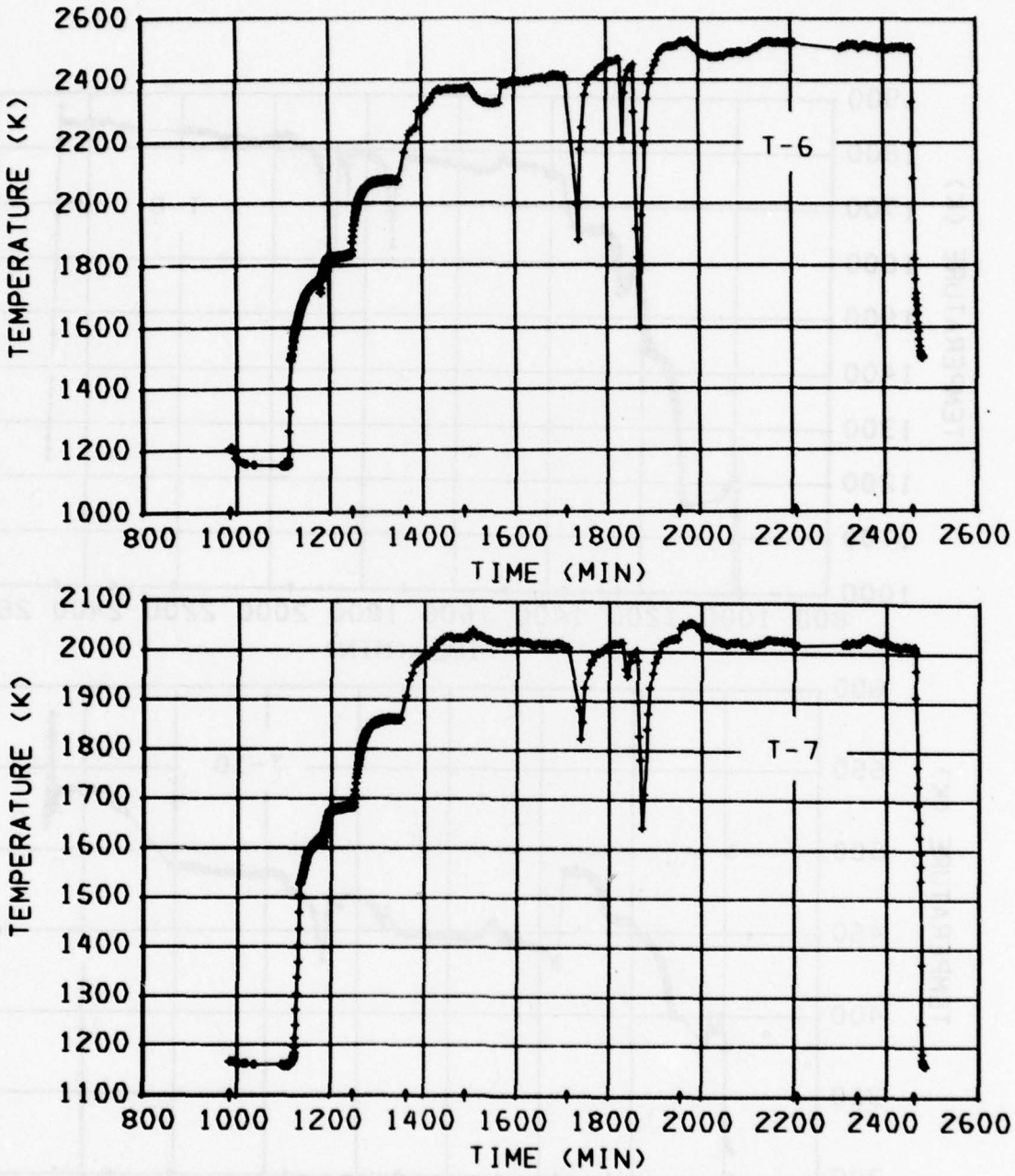

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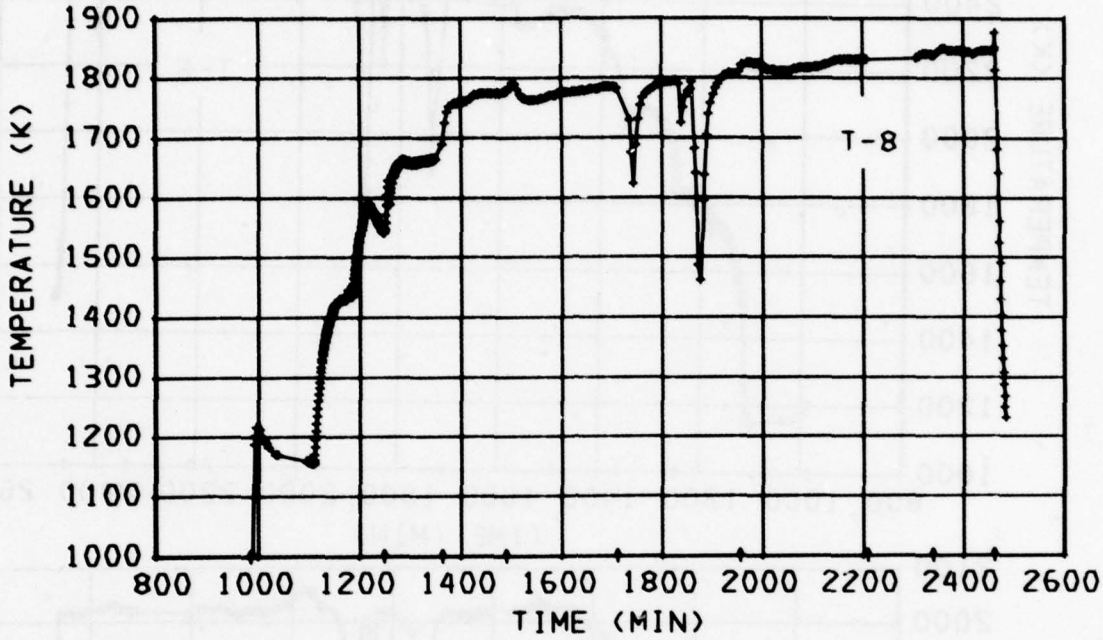

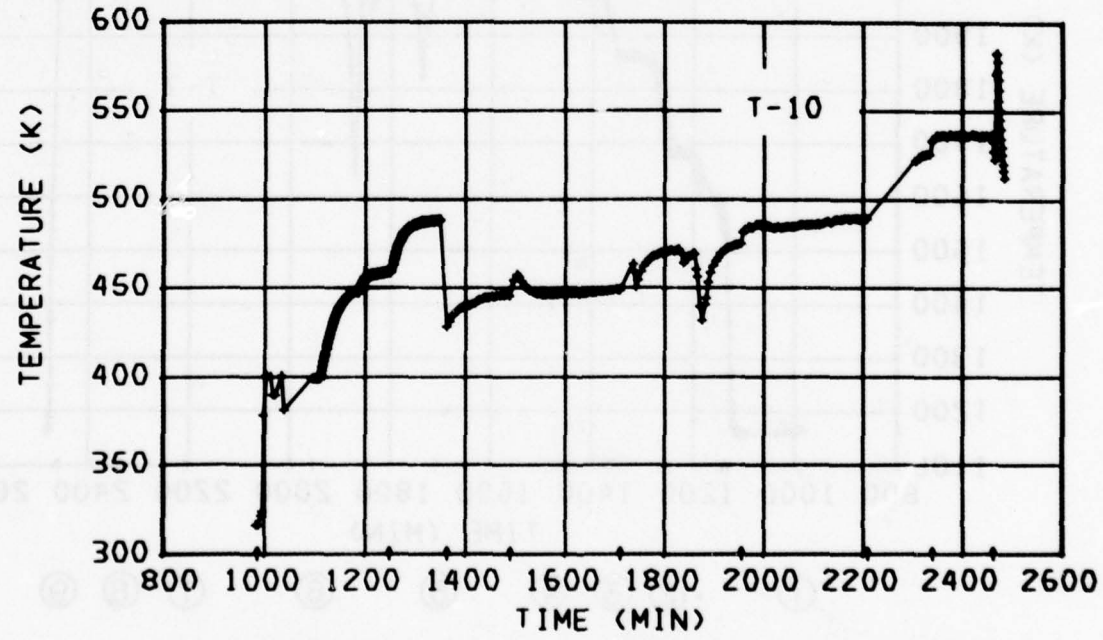

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DAS TIME OR REAL TIME (H)

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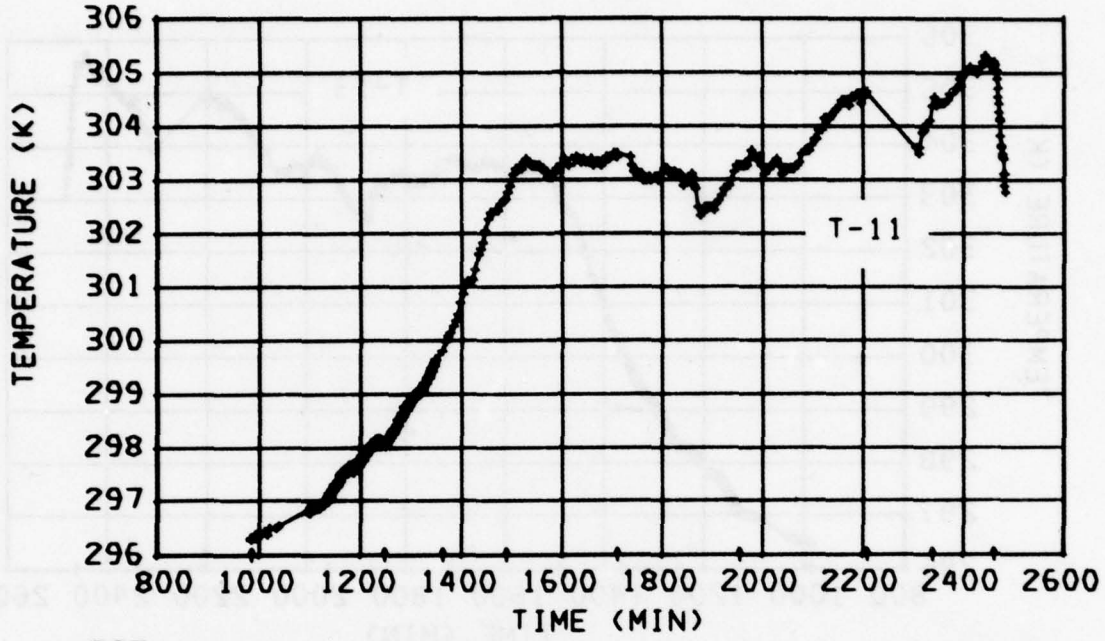

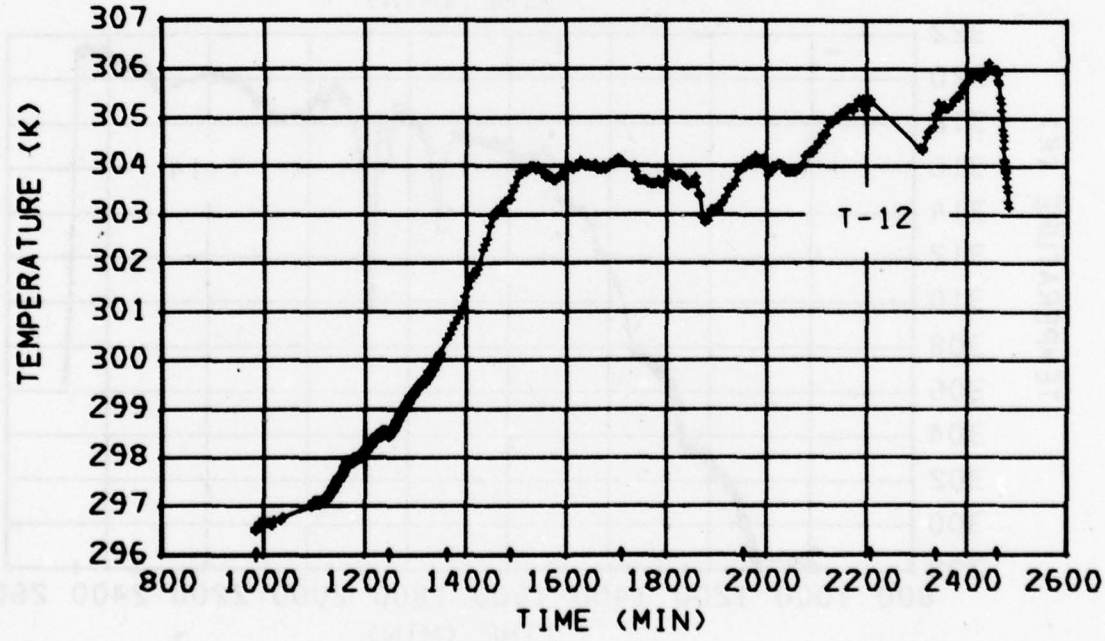

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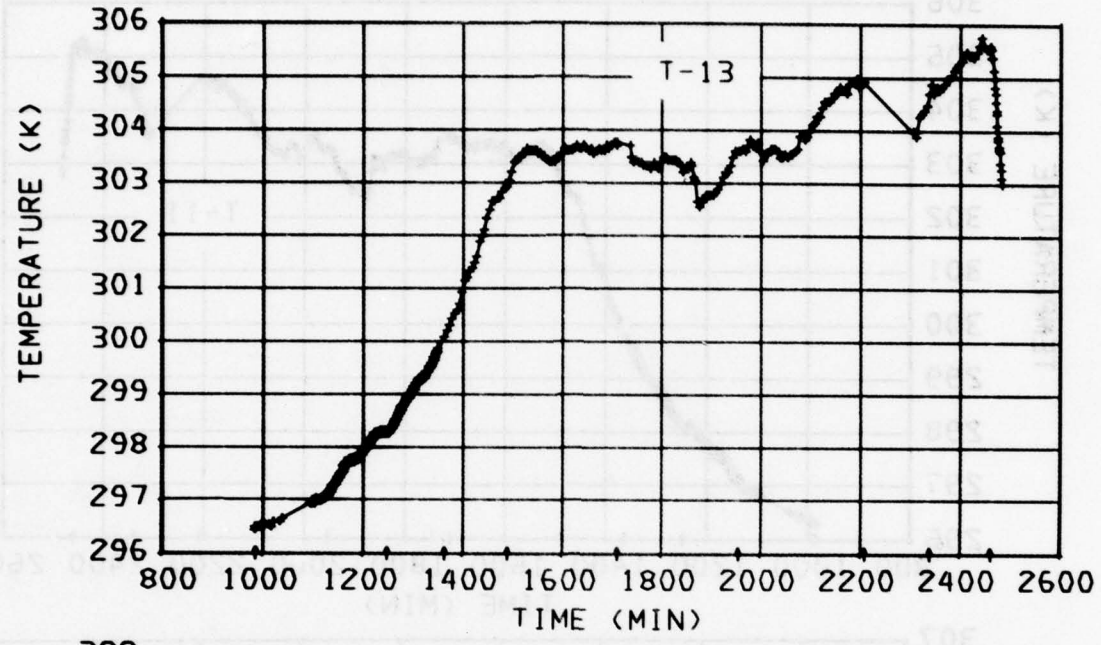

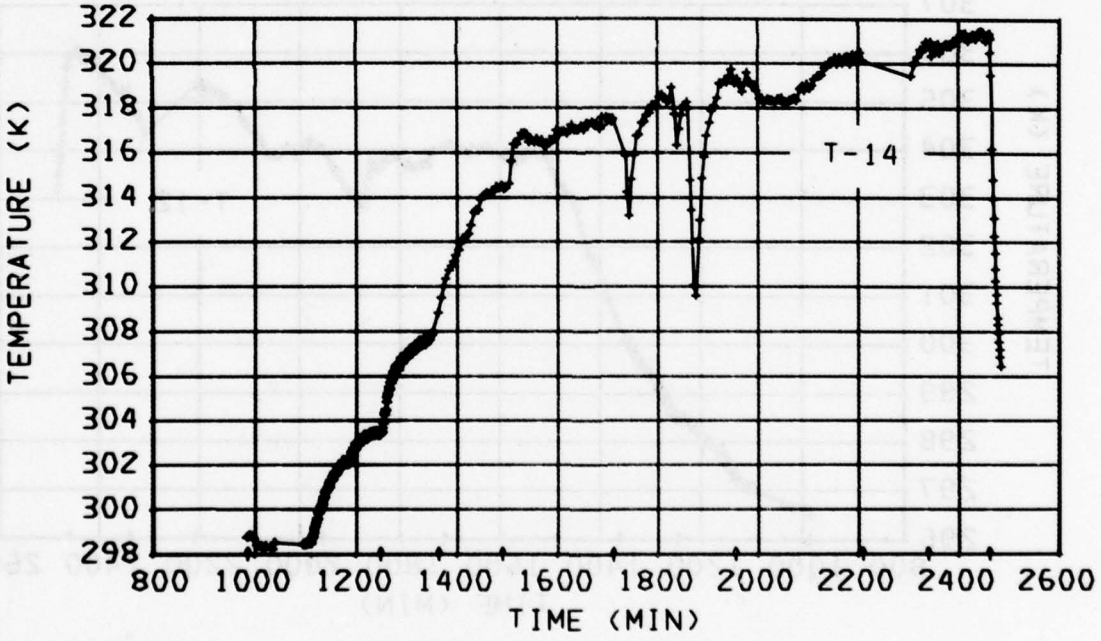

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DAS TIME OR REAL TIME (H)

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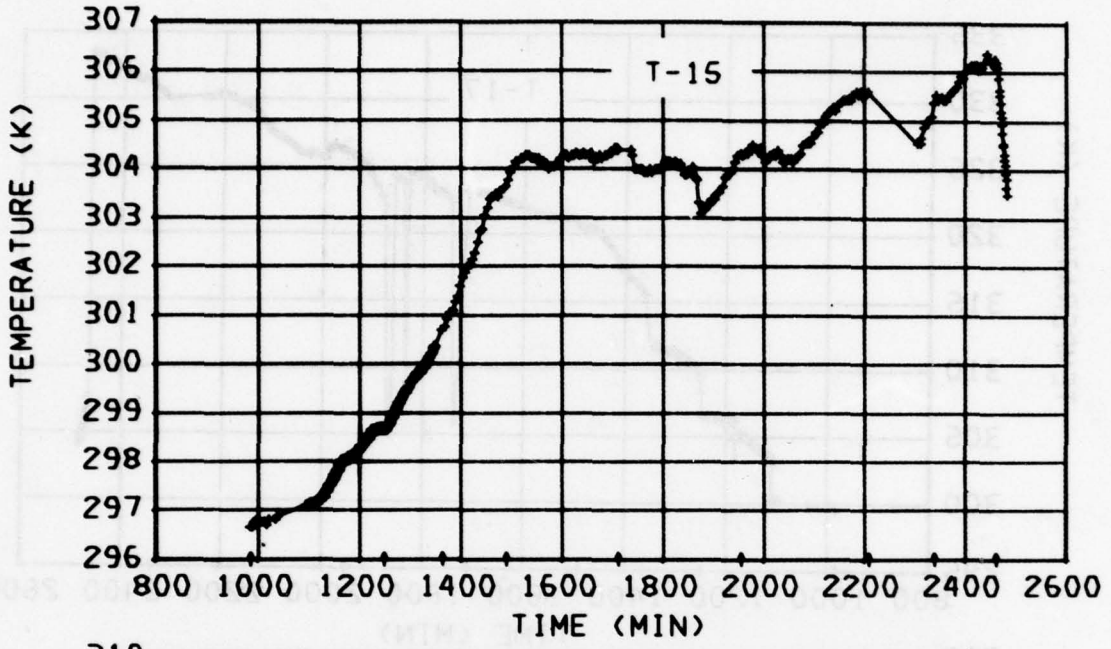

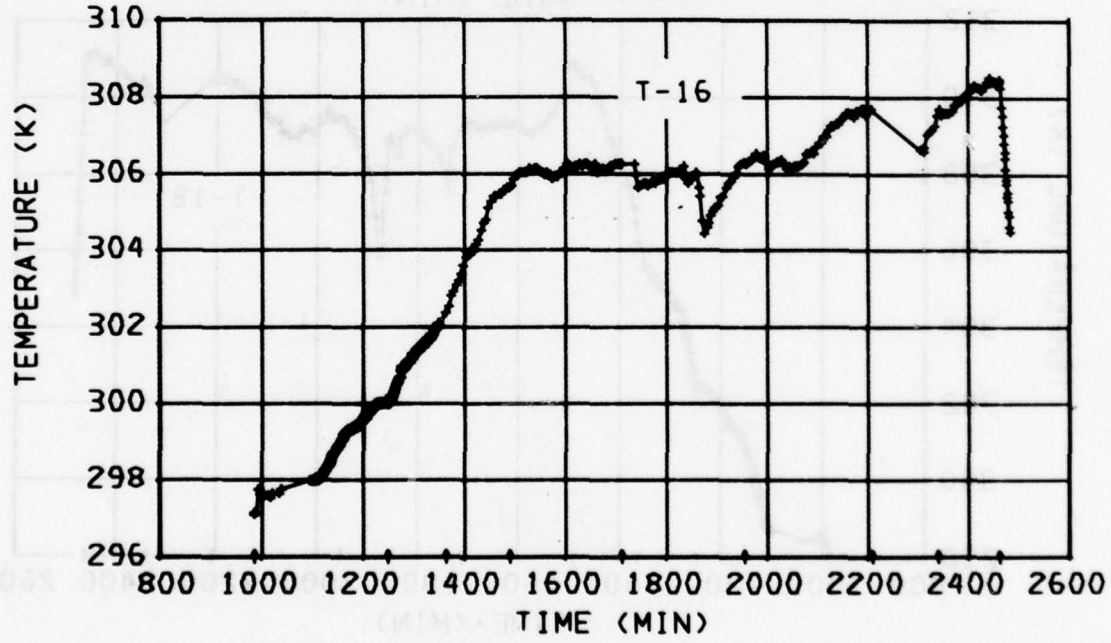

① ②③④ ⑤ ⑥ ⑦⑧⑨

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DAS TIME OR REAL TIME (H)

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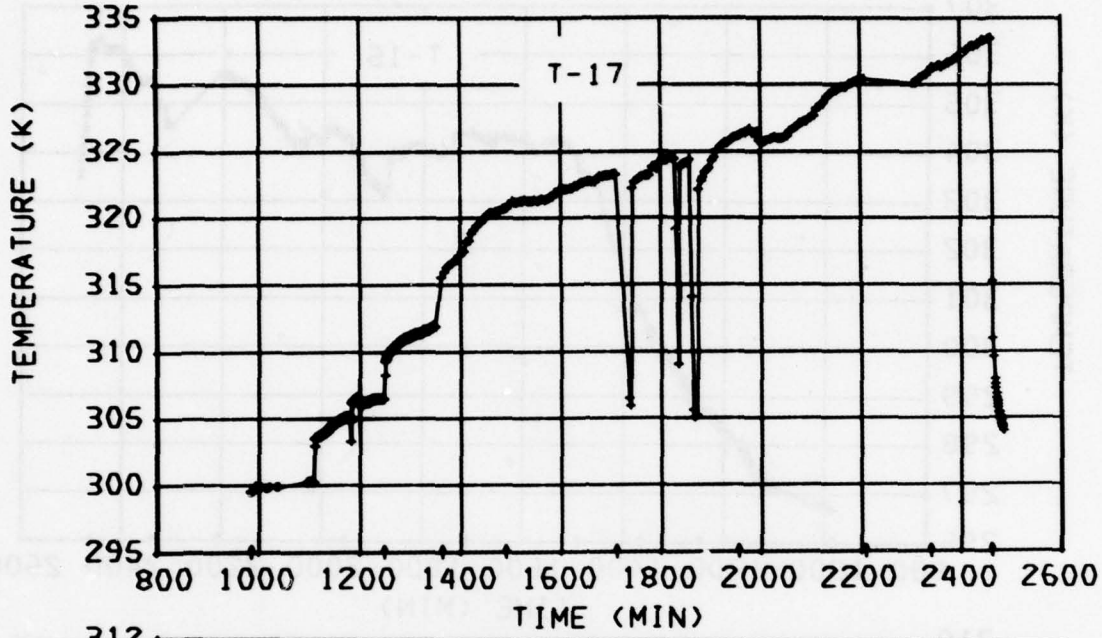

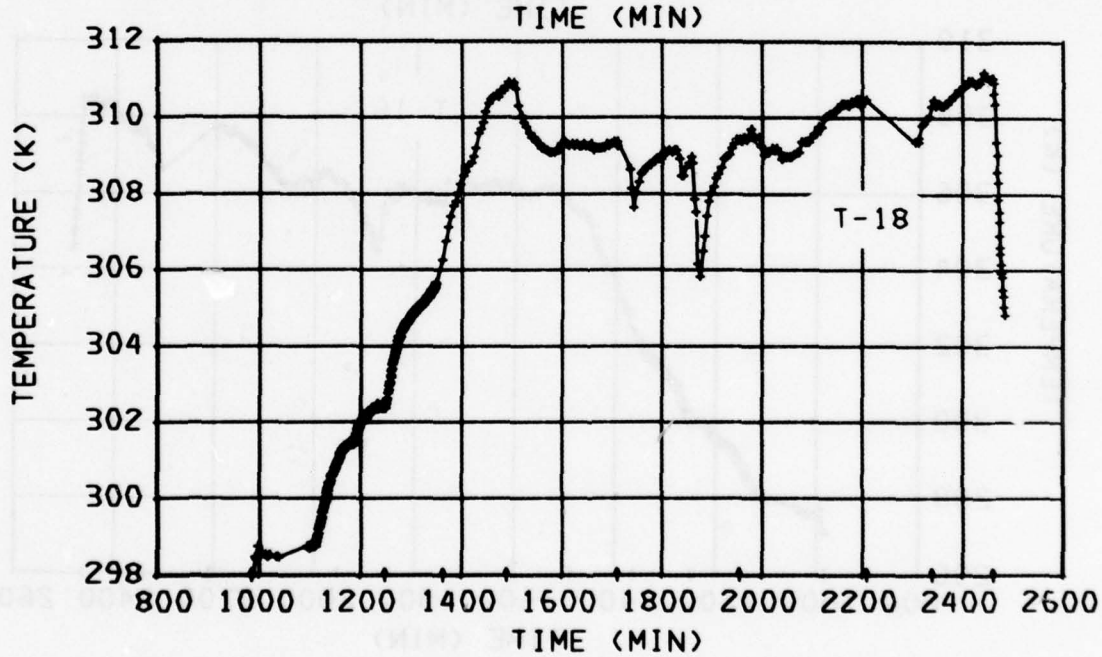

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DAS TIME OR REAL TIME (H)

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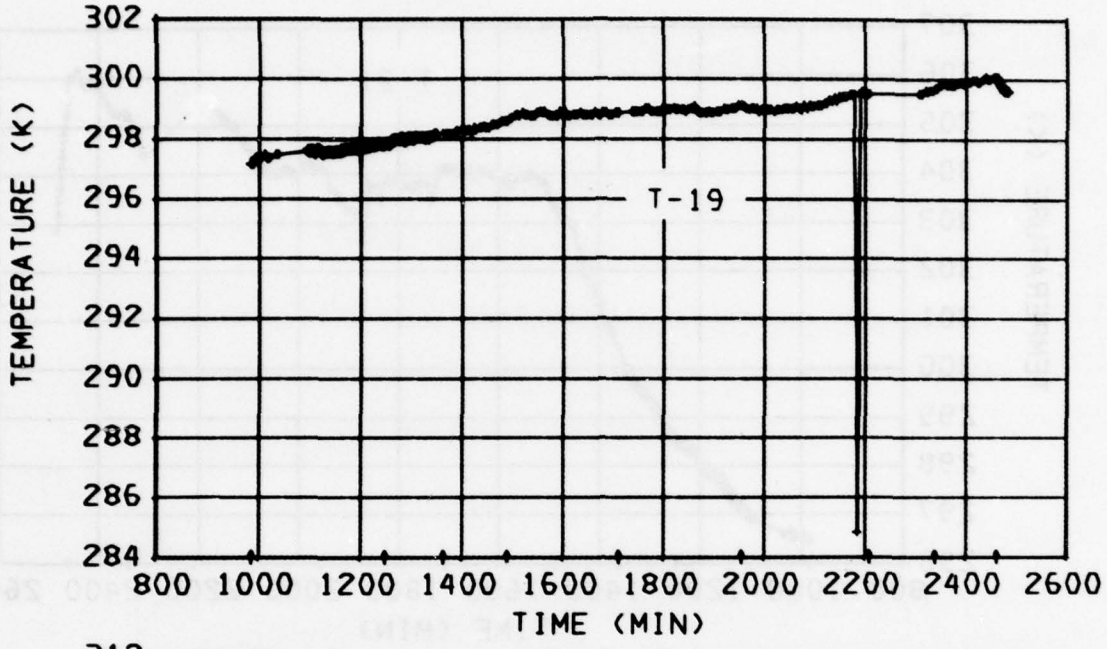

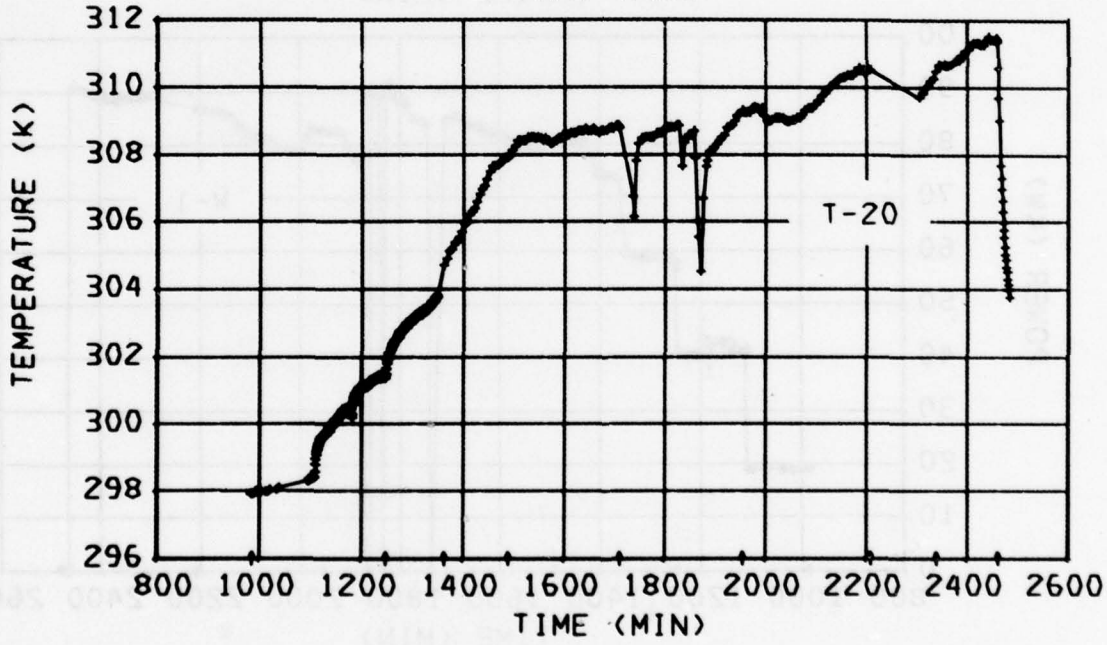

① ②③④ ⑤ ⑥ ⑦⑧⑨

1320 1640 2000 2320 0240 0600 0920 1240 1600 1920

DAS TIME OR REAL TIME (H)

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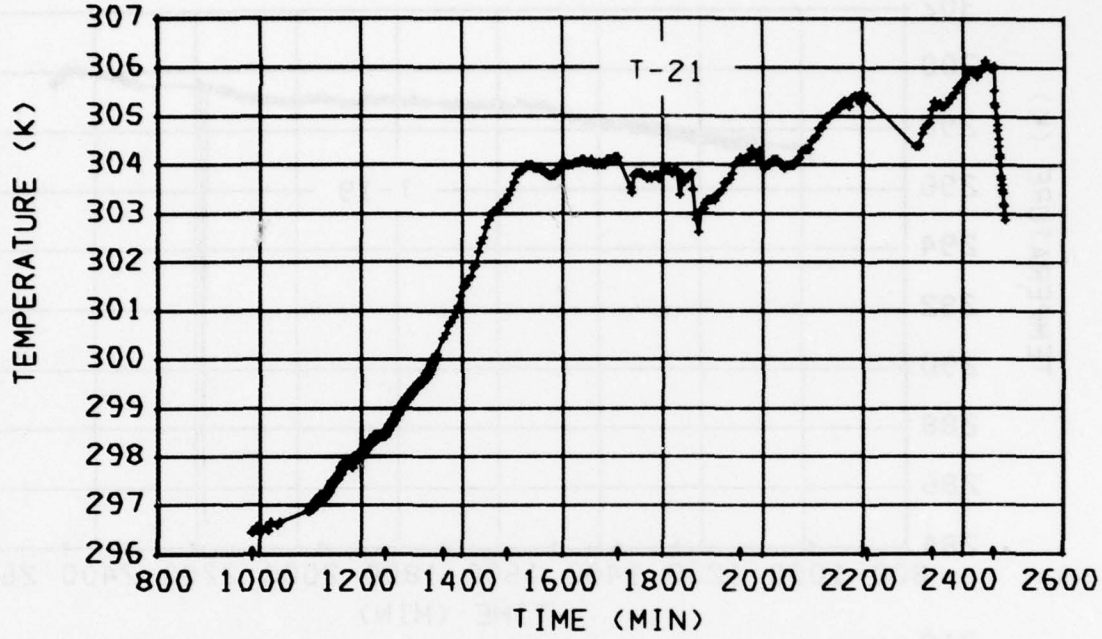

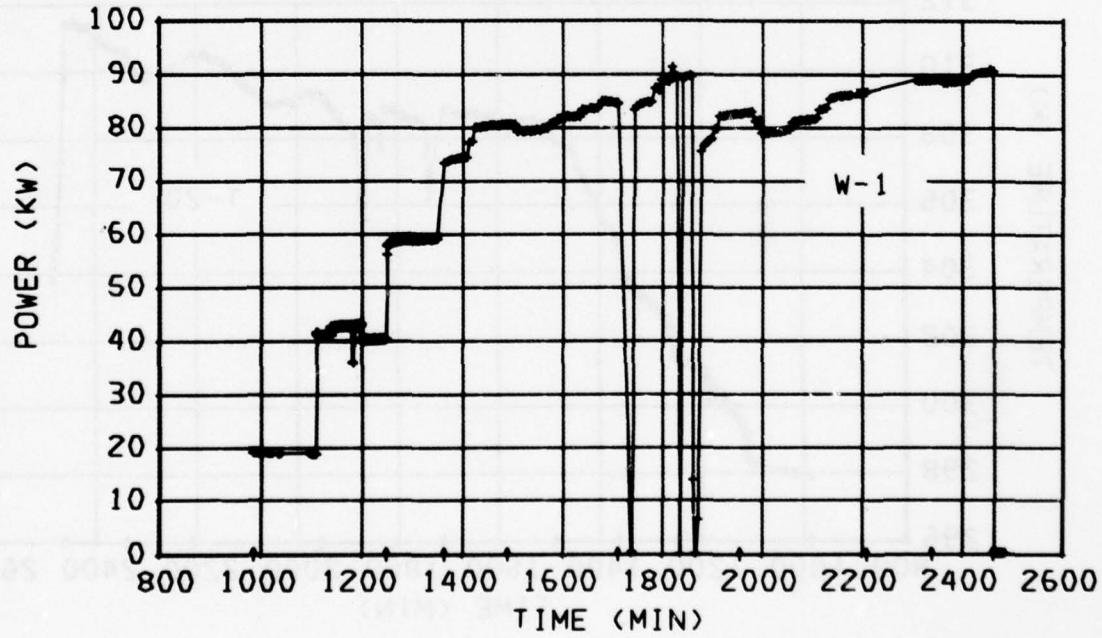

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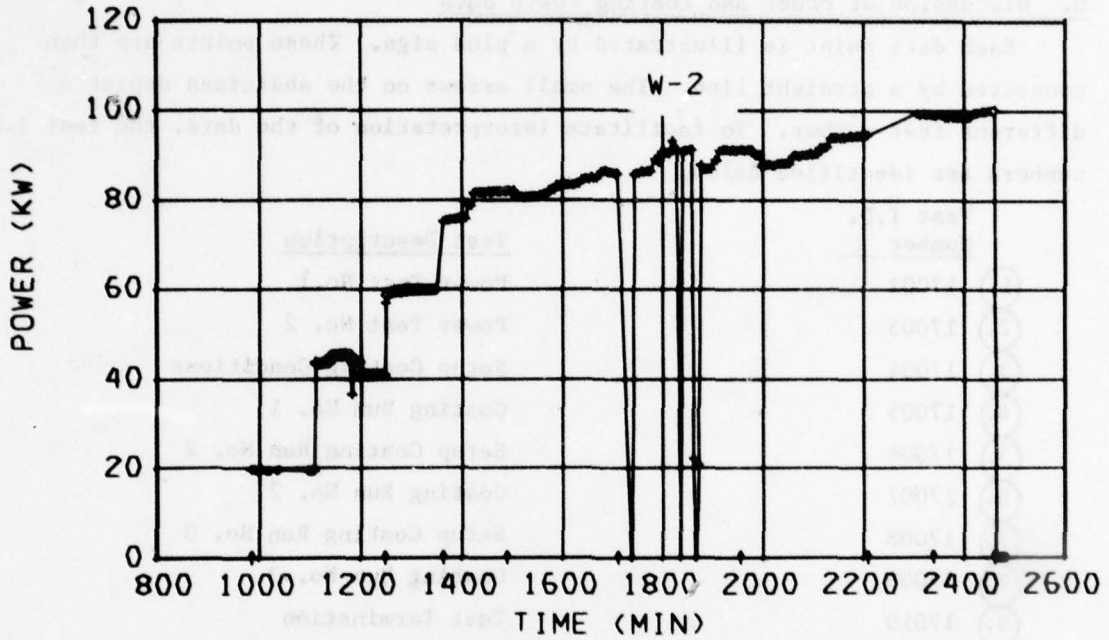
1320 1640 2000 2320 0240 0600 0920 1240 1600 1920

DAS TIME OR REAL TIME (H)

① THRU ⑨: SEE TEXT FOR TEST NO. DESCRIPTION



① ② ③ ④ ⑤ ⑥ ⑦ ⑧ ⑨

1320 1640 2000 2320 0240 0600 0920 1240 1600 1920

DAS TIME OR REAL TIME (H)

① THRU ⑨: SEE TEXT FOR TEST NO. DESCRIPTION

D. Discussion of power and coating tests data

Each data point is illustrated by a plus sign. These points are then connected by a straight line. The small arrows on the absicissa depict a different test number. To facilitate interpretation of the data, the test I.D. numbers are identified below.

	Test I.D. Number	Test Description
1.	17002	Power Test No.1
2.	17003	Power Test No. 2
3.	17004	Setup Coating Conditions
4.	17005	Coating Run No. 1
5.	17006	Setup Coating Run No. 2
6.	17007	Coating Run No. 2
7.	17008	Setup Coating Run No. 3
8.	17009	Coating Run No. 3
9.	17010	Test Termination

The data from the flow meter that measured the nitrogen flow rate (F-1) became erratic at the beginning of Test 17002 (1625 h). The unit had to be turned off periodically to cool the electronics, which explains the large fluctuations. The actual flow rate is the value obtained just before turning off the flow meter (approximately 30 min after it was turned on). the unit was repaired before test series 18000 was run.

The turbine flow meter that measured the cooling water flow rate to the furnace center body (F-7) failed before the test. There are almost no pressure variations in the water supply, so the flow rate through this loop could be determined from the previous calibrations of the water-cooling circuits (by ratios), so we decided to perform the series 18000 tests without this measurement. Even with the flow rate through this loop calculated from the other data, the inaccuracy of this measurement is still less than 2%.

The thermocouple that measured T-3 appears to have become contaminated, and its data are considered inaccurate throughout these series of tests.

The large negative-going excursions in some of the temperature data and the power data were caused by turning off the furnace power to adjust the power factor.

IV. SECOND LASL COATING TEST

The test identification numbers for these tests are in the 18000 series.

The data presented herein are for tests 18003 through 18009.

B. Test log - Second LASL coating test

The original of the test log for test series 18000 is on file at LASL. It outlines pertinent events and their time of occurrence and is enclosed so that the reader may correlate these events with the data graphs.

C. Test Data - Second LASL coating test

The data in the following figures depict the part of the test in which power was applied to the furnace. It was not convenient to present the data taken during other parts of the test in this format, so parts of the data appear in Sec. III. All data were recorded on magnetic tape, which is on file at LASL.

D. Discussion of second LASL coating tests data

Each data point is illustrated by a plus sign. These data points are then connected together by a straight line. The small arrows on the abscissa depict a different test number.

To facilitate data interpretation, the test I.D. numbers are as follows:

	Test I.D. Numbers	Test Description
1.	18003	Setup Coating Run No. 1
2.	18004	Coating No. 1
3.	18005	Setup Coating No. 2
4.	18006	Coating No. 2
5.	18007	Setup Coating Run No. 3
6.	18008	Coating No. 3
7.	18009	Test Termination

The turbine flow meter that measured the cooling water flow rate to the furnace center body (F-7) failed again before these tests. We decided to proceed with the tests for the reasons outlined previously.

The thermocouple that measured parameter T-3 apparently failed before the tests, so these data are considered inaccurate throughout these series of tests.

SERIES 18000 TEST LOG

DATE: 11-4-76 Day 309 TEST: 18000-18009

TIME	REMARKS	F/G
153000	Test ID to 17000	
164130	Completed water flow measurement #1 Per paragraph 2.6.17 of test procedure dated October 1976. File gap	F/G
164400	Completed water flow measurement #2 Per paragraph 2.6.29 of test procedure dated October 1976. File gap	F/G
164630	Completed water flow measruement #3 Per paragraph 2.6.39 of test procedure dated October 1976. File gap.	F/G
165000	Test ID to 18000 Initiated 1 h scans and printout. Furnace power on	
	Day 310	
155111	Turned furnace power off. Made 10 data scams/ File gap File gap	F/G F/G
160000	Turned DAS off and placed BOT marker on data tape	
	Day 313	
101200	Test ID to 18000 Made 10 data scans with furnace power off.	
101500	Initiated 5 min scans and printout	
102100	Turned DAS printer off.	
124500	Discontinued DAS scan. Looking for pressure leak in furnace	
125000	Initiated 1 h scans	
134500	Still on 1 h scan. Starting test procedure. Leak isolated to thermocouple feed thru area.	
141900	Set N_2 flow to 3.4166 SCFM	

SERIES 18000 TEST LOG (cont'd)

142000	Initiated 5 min scans.	
143000	First mass spectrometer scan at 9 V/o CH_4	
145600	Second mass spectrometer scan at 6 V/o CH_4	
151300	Third mass spectrometer scan at 3 V/o CH_4	
152900	Fourth mass spectrometer scan at 1 V/o CH_4	
154700	Fifth mass spectrometer scan at 0 V/o CH_4	
160600	Discontinued 5 min scans File gap	F/G
160630	Set test ID to 18003	
161500	N_2 Flow set to 10 SCFM	
162000	Initiated 5 min scans	
164200	Placed T-6 and T-7 pyrometers in "Run" mode	
164300	Turned furnace power on to 45 kW	
164500 to 171500	Adjusting power factor	
173000	Reversed leads on T-5 thermocouple	
185200	Added 1.0 uf to furnace capacitor. Set power to 40%, 60 kW by control meter.	
192200	Increased power to 63 kW.	
193700	Reduced power to 62 kW	
194500	T-7 stabilized	
200700	Discontinued DAS scan File gap Test ID to 18004	F/G
201000	Initiated 5 min scan and print out	
202500	Missed scan	
202530	Made DAS scan	

SERIES 18000 TEST LOG (cont'd)

204600	Reduced power by 1.5 kW	
211455	Increased power by 1.0 kW	
212530	Increased power by 1.5 kW	
214400	Noticed mist or vapor through T-6 sight glass	
215200	Increased power by 1 kW	
221200	Increased power by 1 kW	
222700	Increased power by 1.5 kW	
223200	Opened roughing pump valve slightly to try and clear vapor from furnace. No help.	
223500	Missed scan. Had pressure readout on DAS in single point mode.	
223600	Made DAS scan	
224500	Back to 5 min scans	
224600	Unable to increase furnace power. Control is limited	
225730	Furnace power off. Added 1.7 uf to capacitor. Furnace power back on to 50%. T-7 is down to 1600°	
231000	Reduced power 1 kW	
235100	Increased power 1 kW	
001400	Power limited, cannot increase	
003000	T-7 temperature still decreasing	
010108	File gap. Test ID to 18005	F/G
010211	Initiated 5 min scans	
010500	Discontinued DAS scan File gap Test ID to 18006	F/G
010800 to 011330	Adjusting furnace power factor	
011400	Furnace power on	

SERIES 18000 TEST LOG (cont'd)

012100	Changed T-6 to high range File gap	F/G
014015	Furnace power off added 1.1 uf to capacitor	
014055	Furnace power on	
014827	Furnace power off. Added 1.0 uf to capacitor	
015700	Furnace power off. Added 2.3 uf to capacitor	
015730	Furnace power on	
021500	Temperature stabilized	
021814 to 61500	Several power adjustments during coating run	
061530	File gap. Test ID to 18007	F/G
062800	File gap. Test ID to 18008	F/G
063100	Decreased power 1 kW	
NOTE:	From 0642 thru 0715 DAS channels 00-04 were not recorded.	
065000	Reset DAS to 5 min scans	
065010	Increased power 3 kW	
073400	Adjustment on CH_4 flow rate	
074700	Changed CH_4 bottle. Initiated scan and printout. Adjustment made on CH_4 flow rate.	
075000	Back on 5 min scans	
075100	Incrased power by 1 kW	
083000	Increased power by 1 kW	
083500	CH_4 supply bottle regulator sticking. Will not maintain supply pressure	
090100	Changed CH_4 supply bottle	

SERIES 18000 TEST LOG (cont'd)

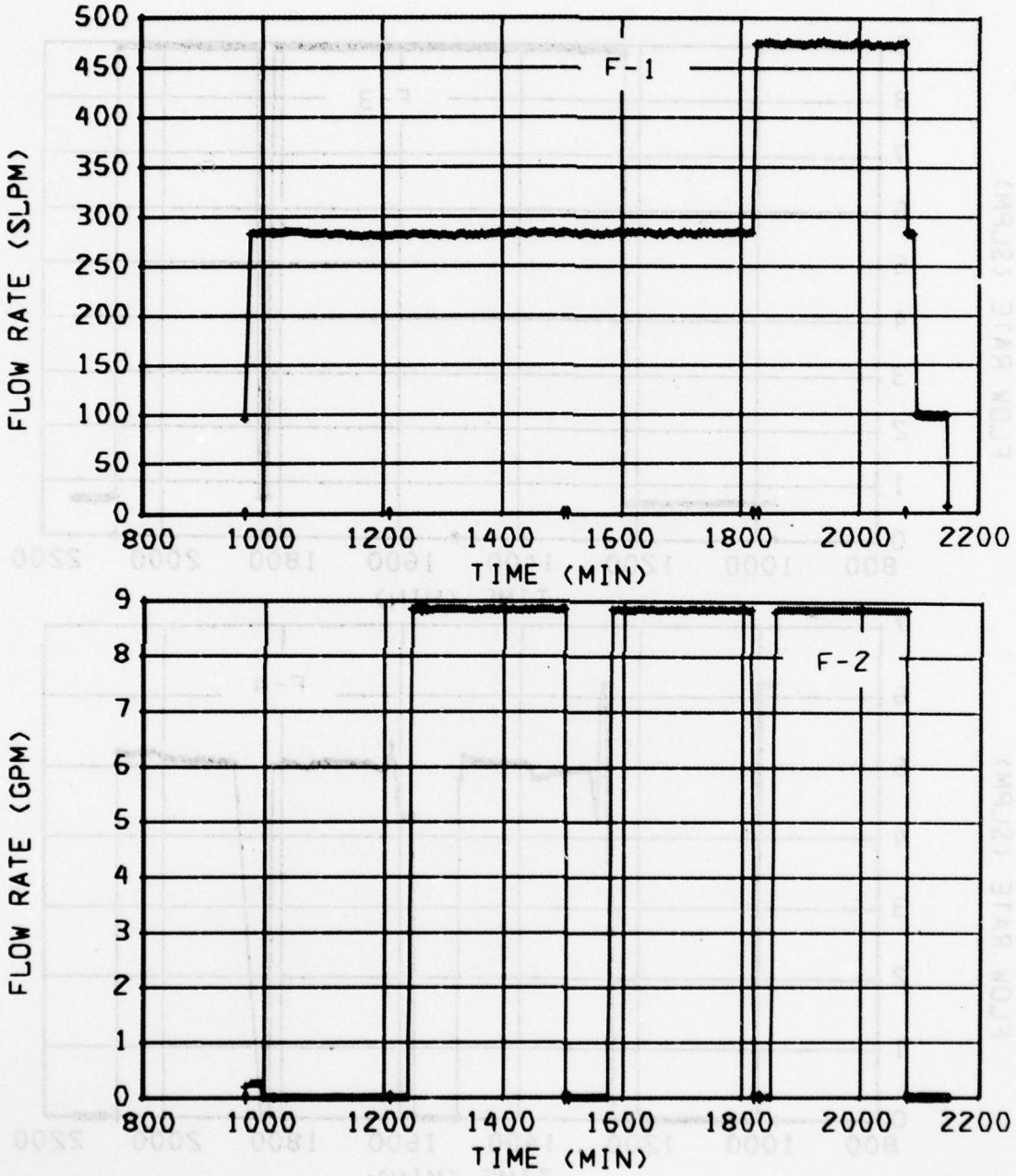
090400	Increased furnace power by 2 kW	
090500	Missed scan while adjusting CH_4 flow rate	
090650	Made scan and printout	
091200	Readjusted CH_4 flow	
091500	Back on 5 min scans unable to increase power to furnace.	
103600	File Gap. Test ID to 18009	F/G
103900	Setting N_2 flow rate	
104000	Initiated 1 min scans	
105130	T-7 pyrometer to low range File gap N_2 flow rate reset	F/G
105530	Back on 1 min scans	
105955	T-7 pyrometer to "CAL" mode	
110445	T-6 pyrometer to "CAL" mode	
114545	Discontinued 1 min scans File gap	F/G
114630	10 data scans at zero conditions File gap	F/G
120000	File gap	F/G

Total 17 file gaps
15 files

REDUCED DATA FOR SECOND COATING TEST

SERIES 18000

The reduced test data are ploted versus normalized time in minutes. A second reference scale at the bottom of each page gives the corresponding DAS real time hours. The label on each figure identifies the parameter plotted (see Table B-1). The parameters are arranged in alphabetical and numerical order.



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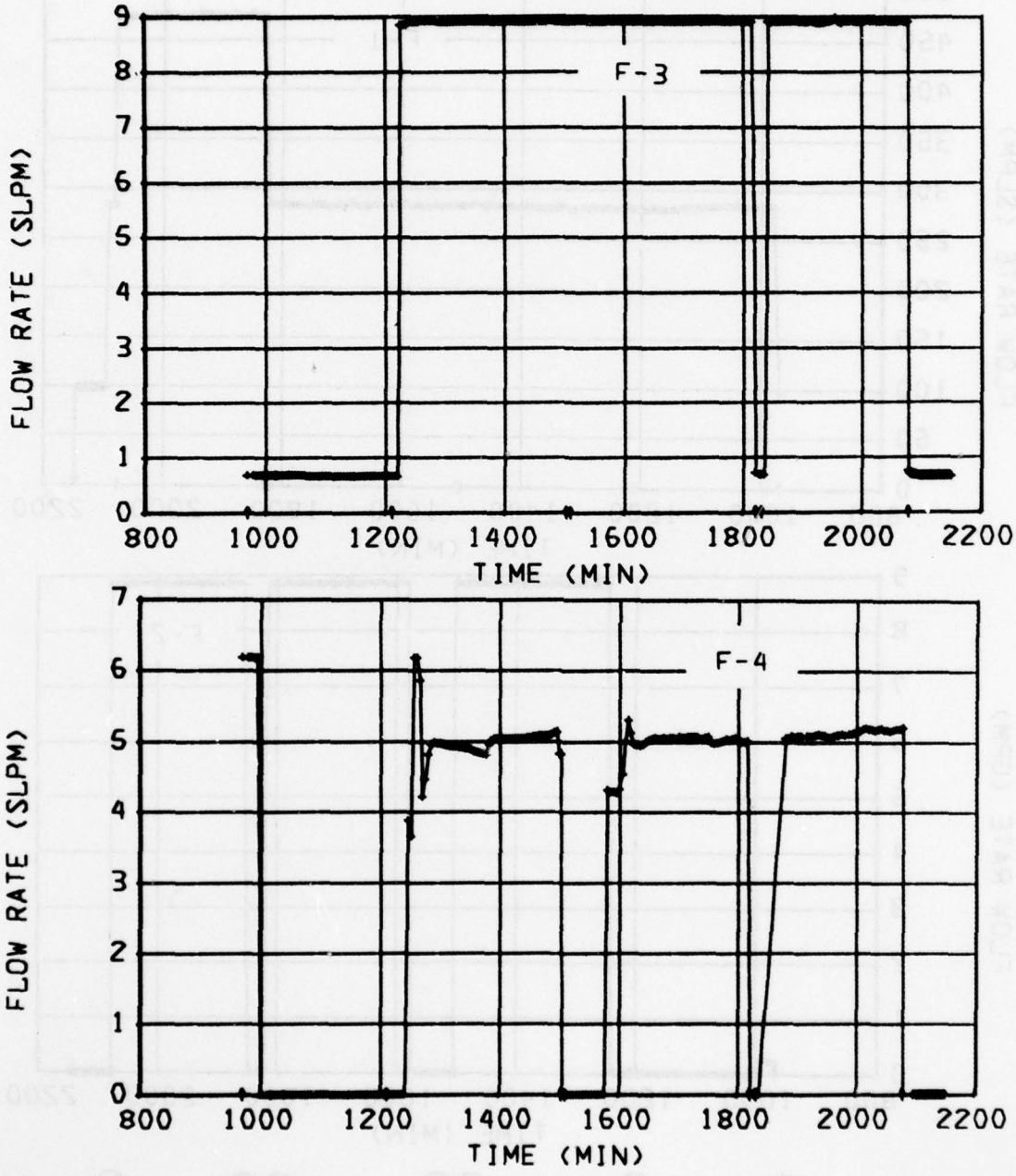

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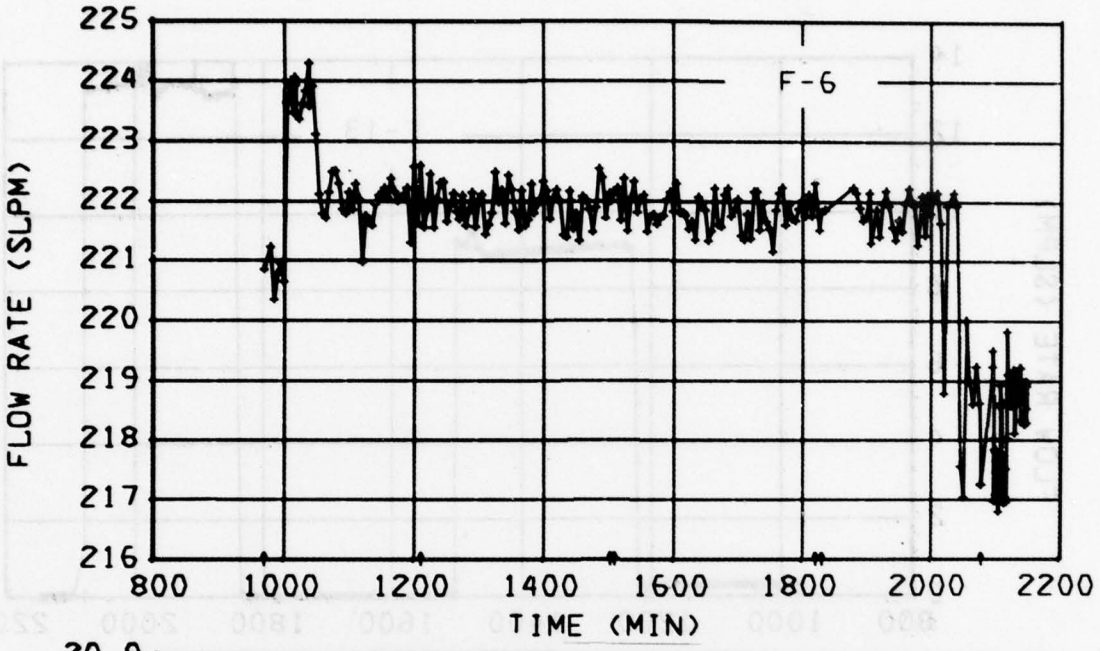

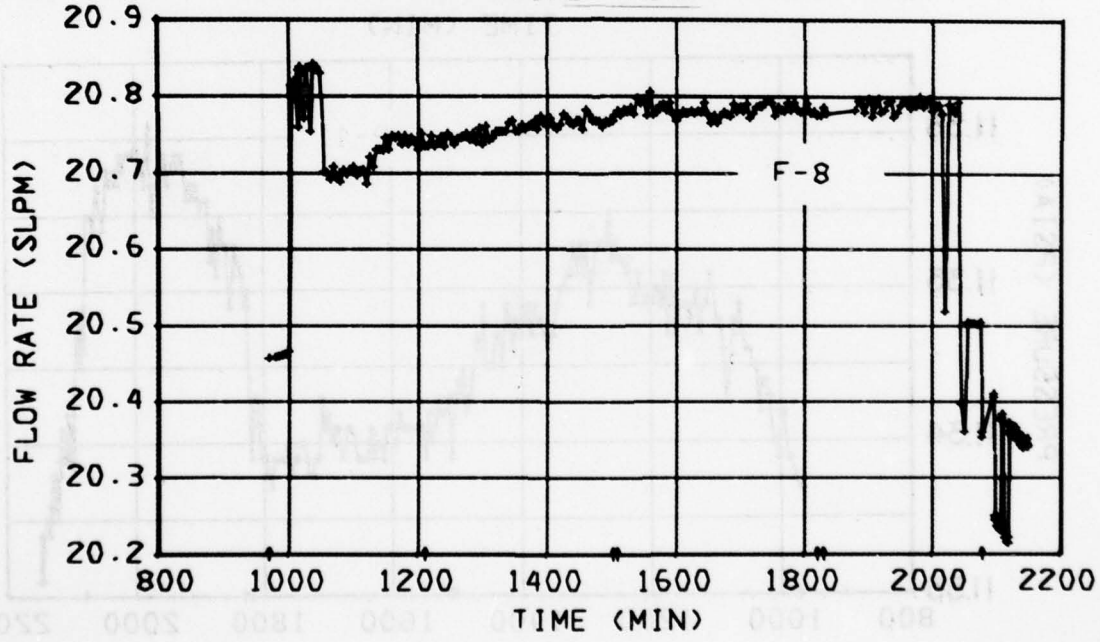

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DAS TIME OR REAL TIME (H)

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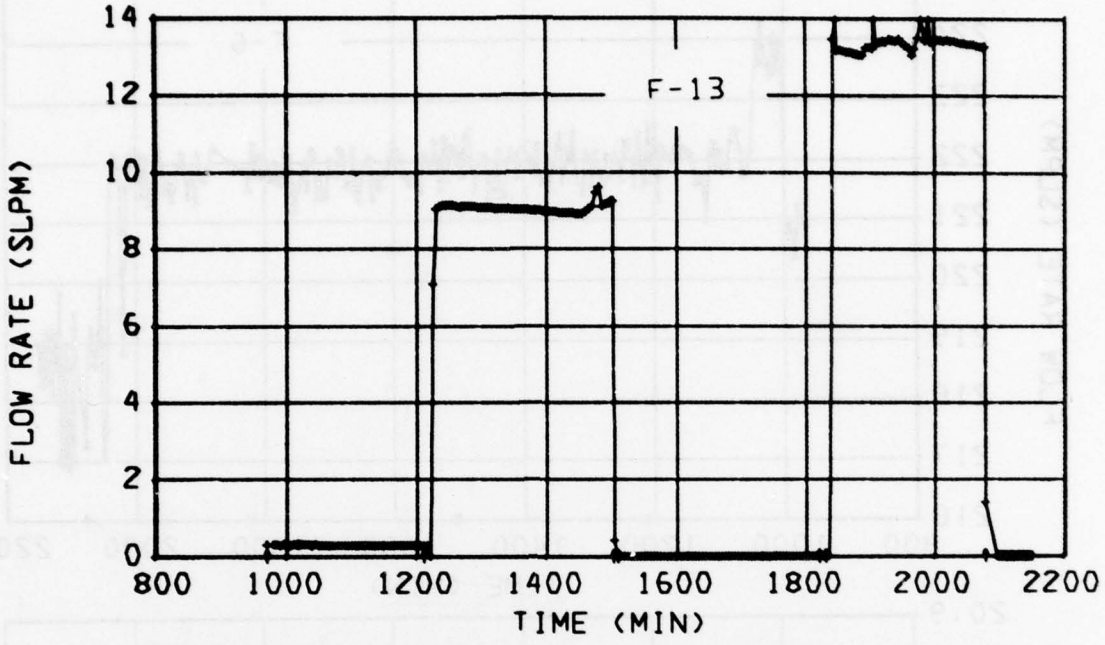

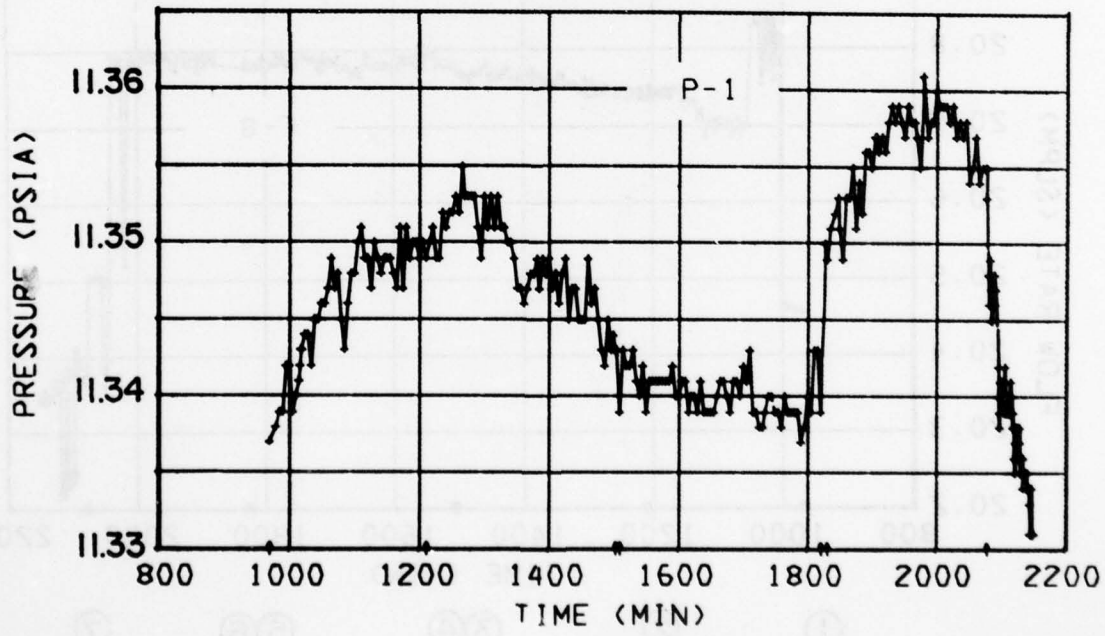

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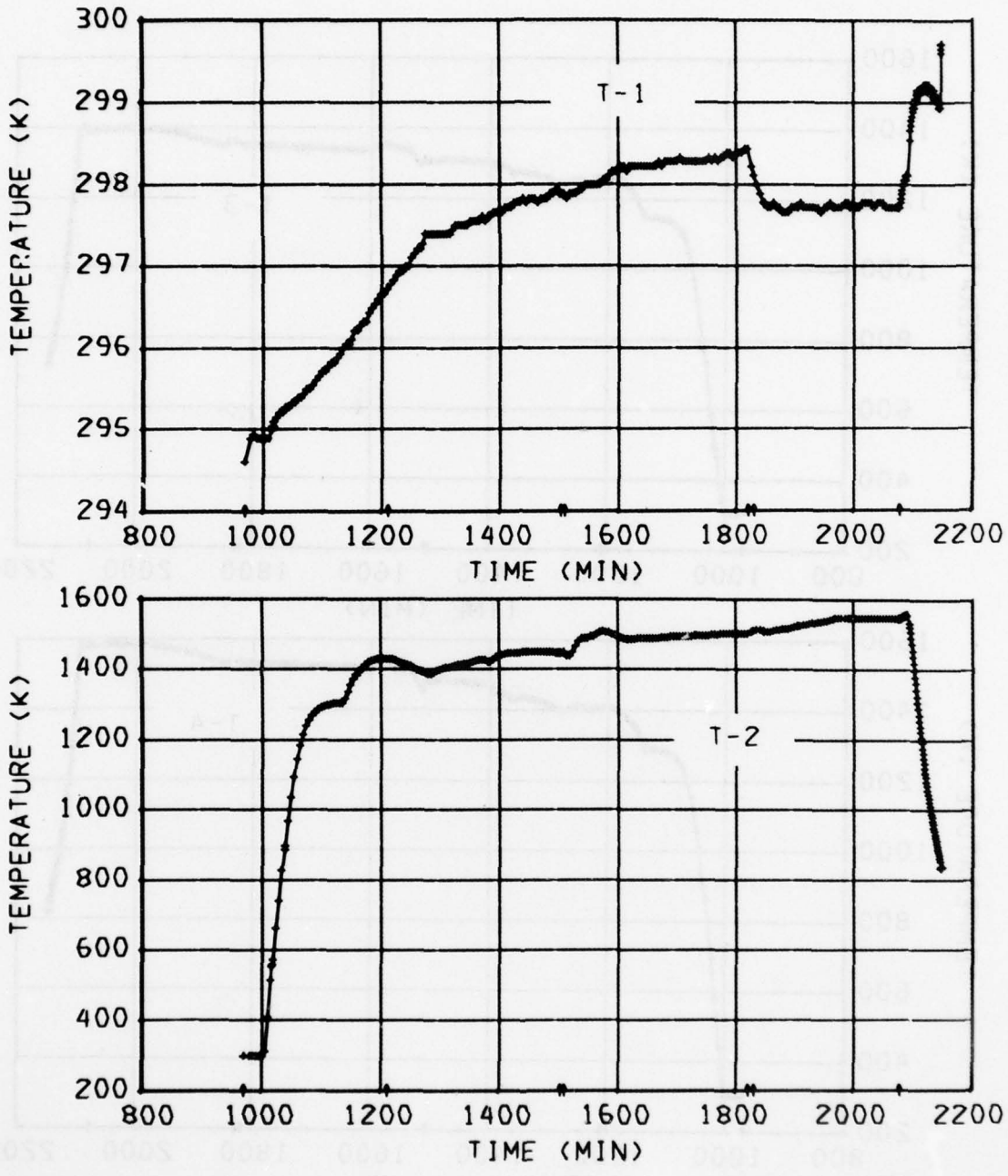

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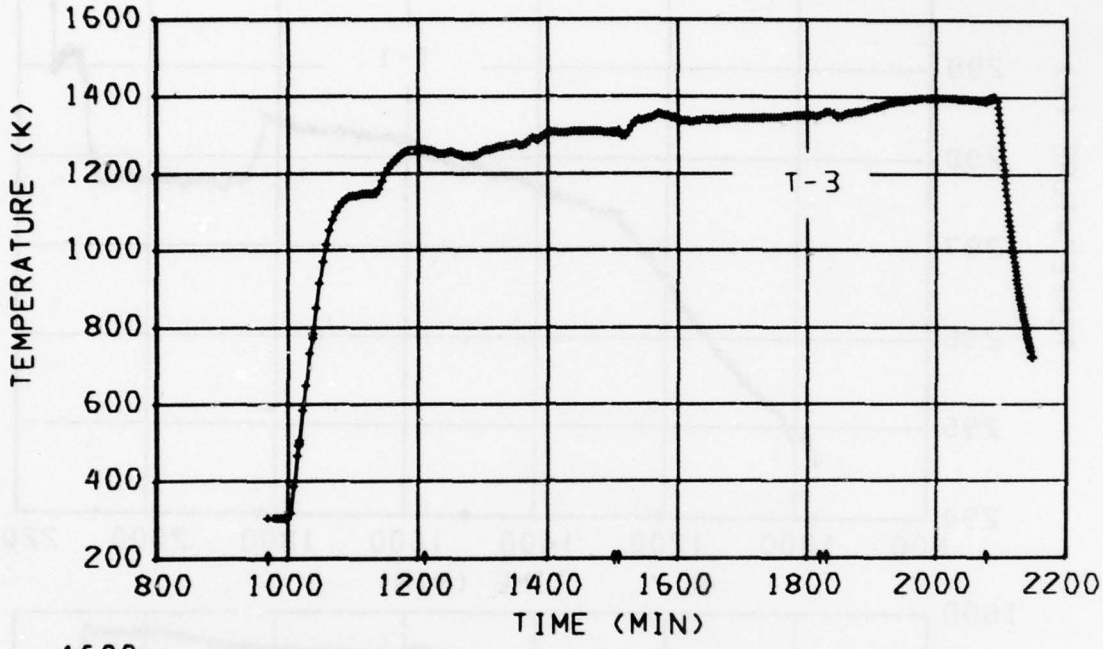

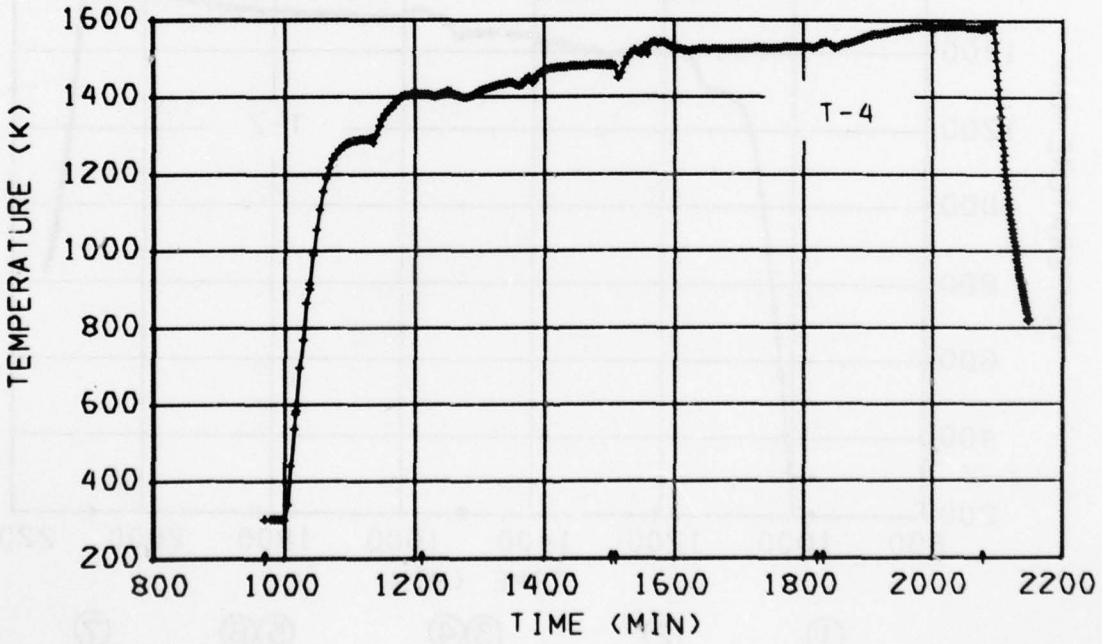

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DAS TIME OR REAL TIME (H)

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JUN 78 T C WALLACE, G E CORT, J J DAMRAN F04611-76-X-003
UNCLASSIFIED AFRPL-TR-78-46-VOL-2 NL

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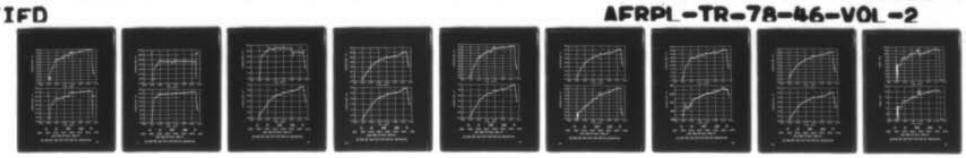

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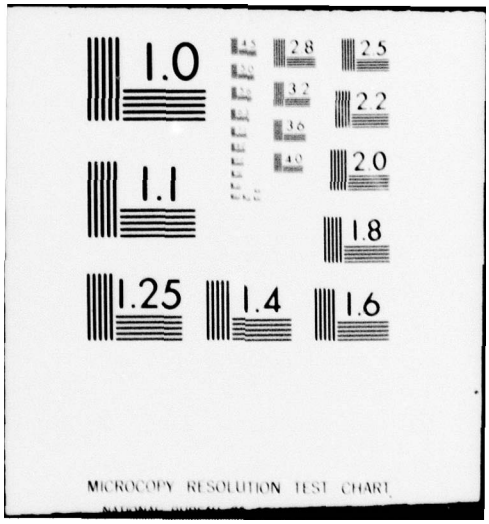
1.0
1.1
1.25
1.4
1.6
1.8
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2.2
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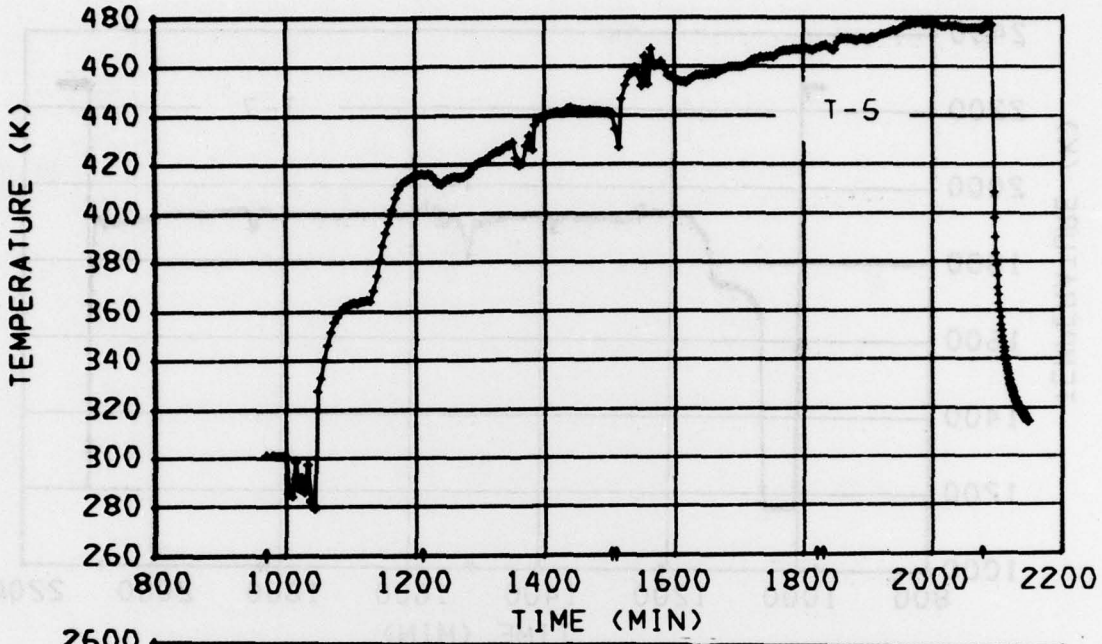
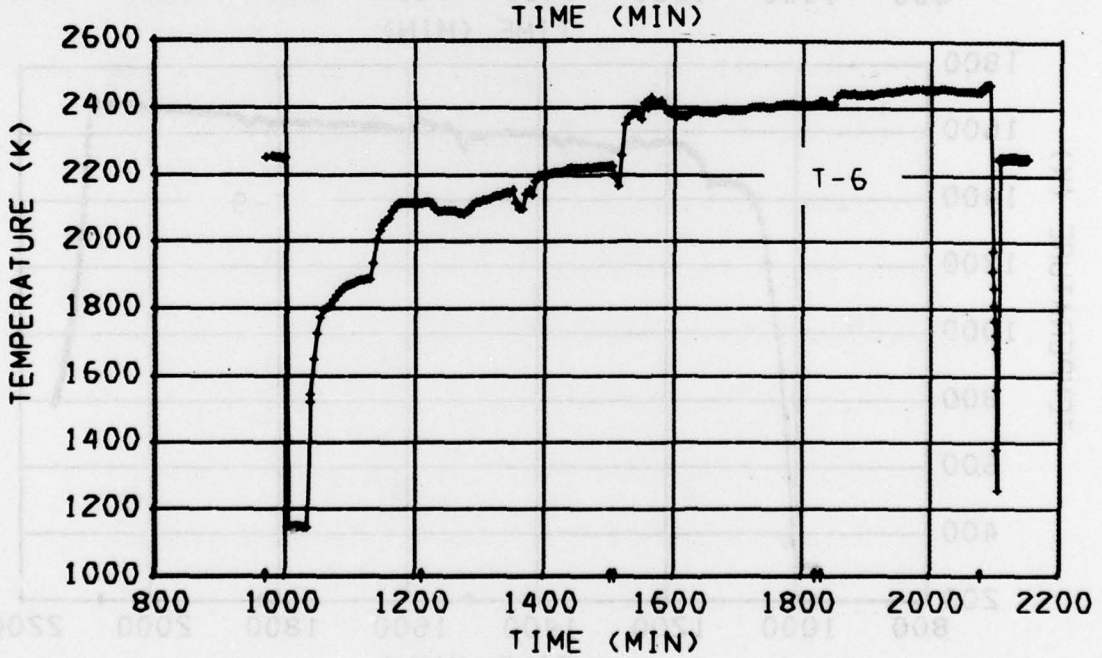
3.2
3.6
4.0
MICROCOPY RESOLUTION TEST CHART





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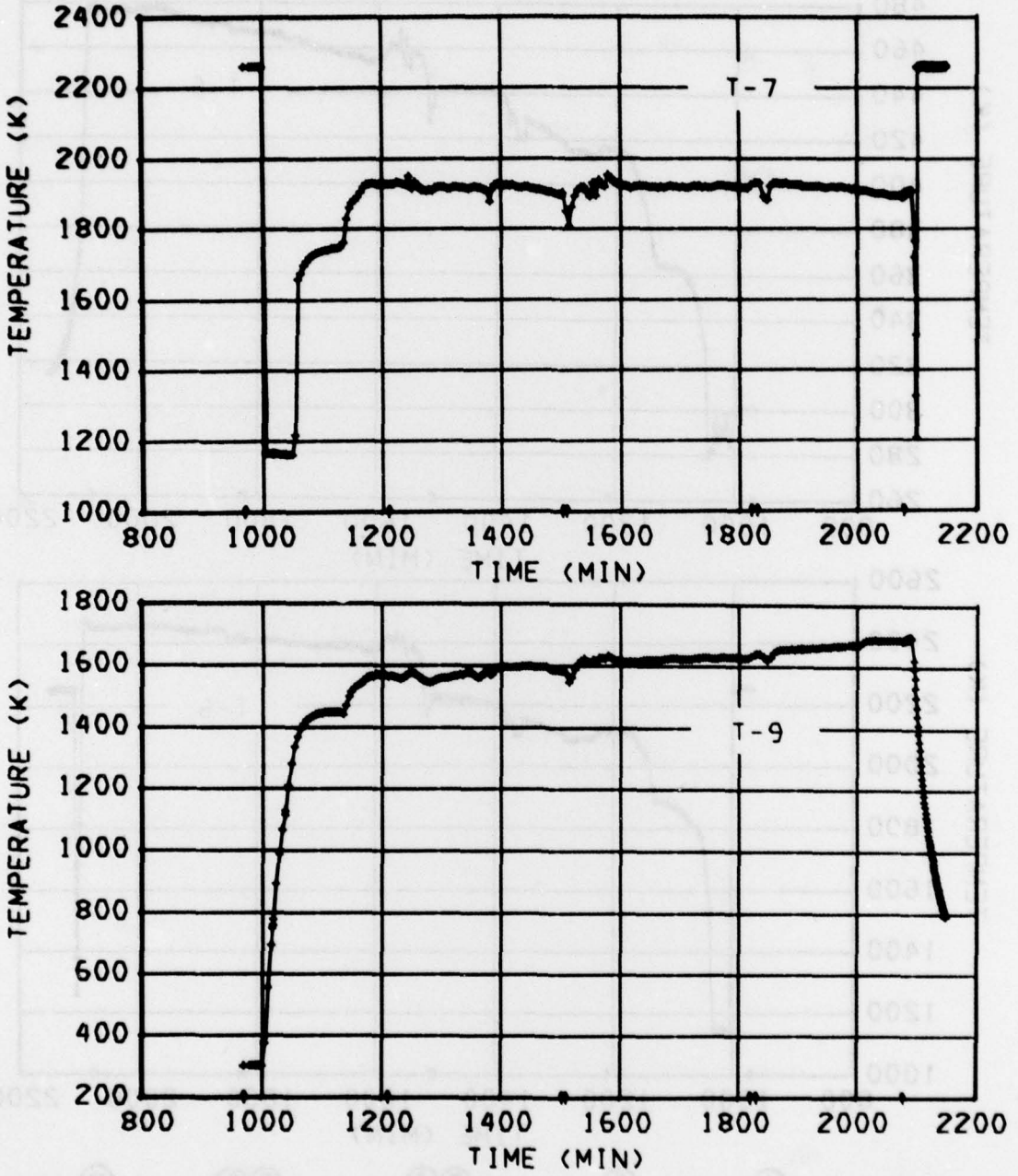

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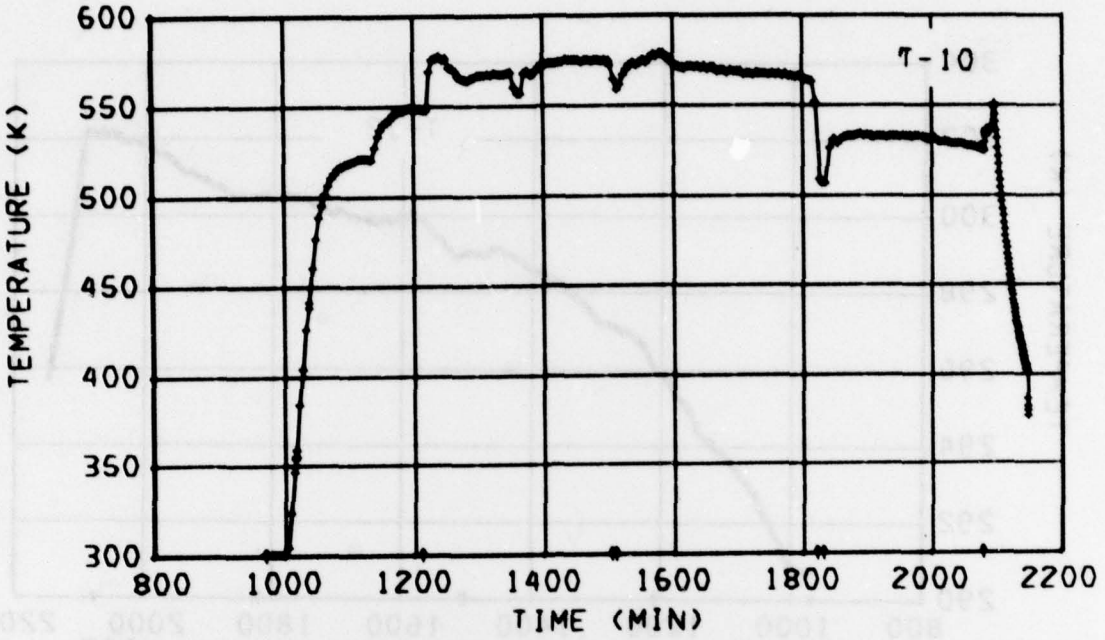

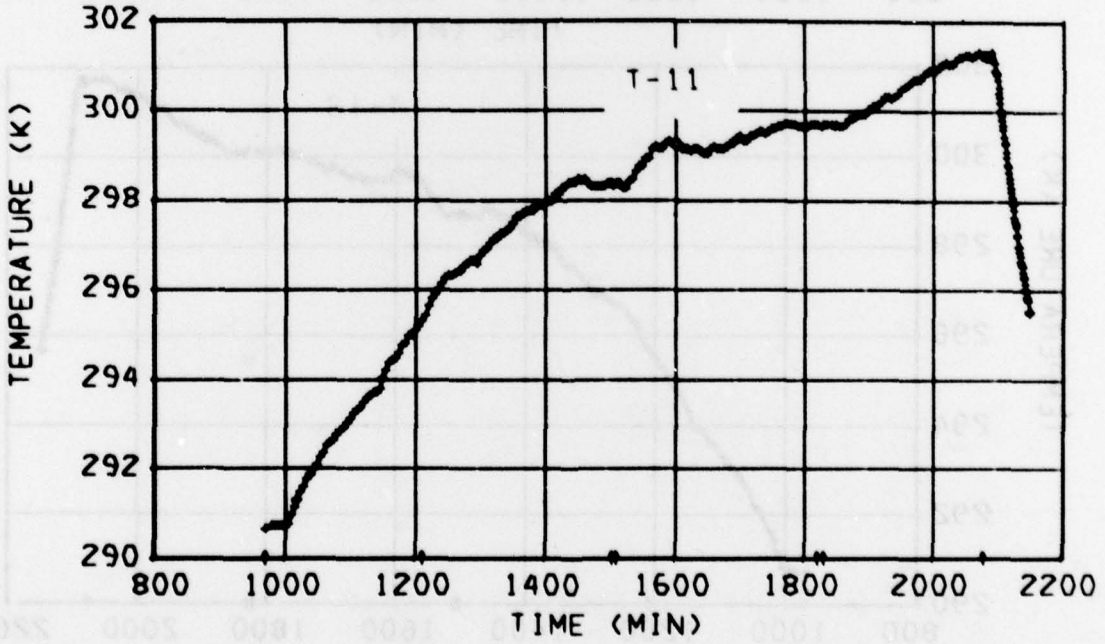

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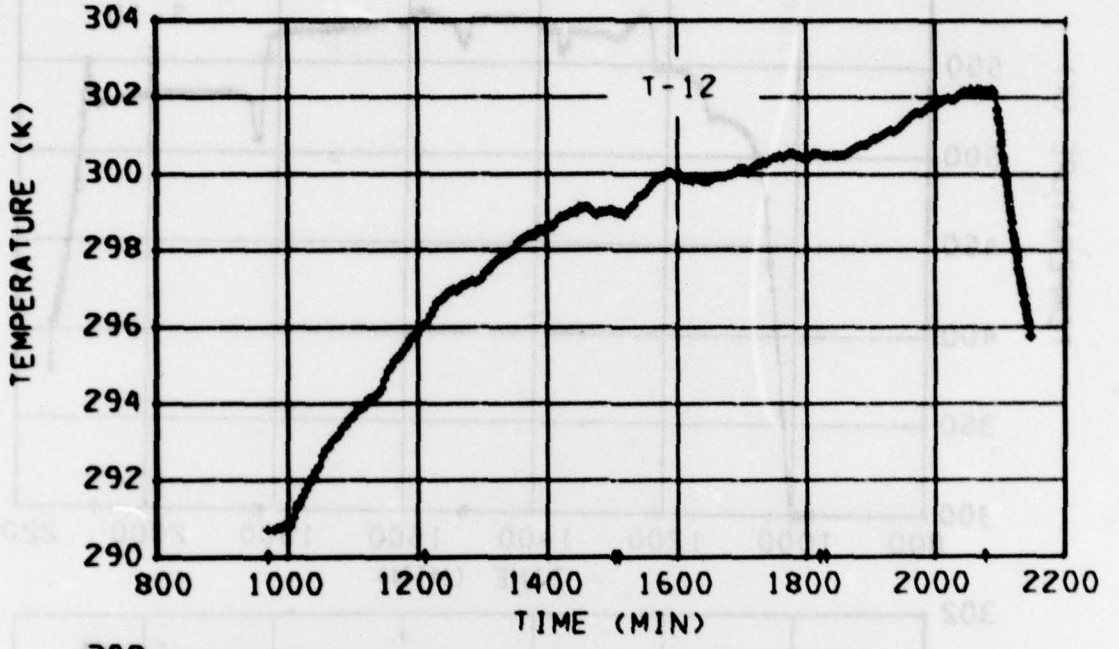

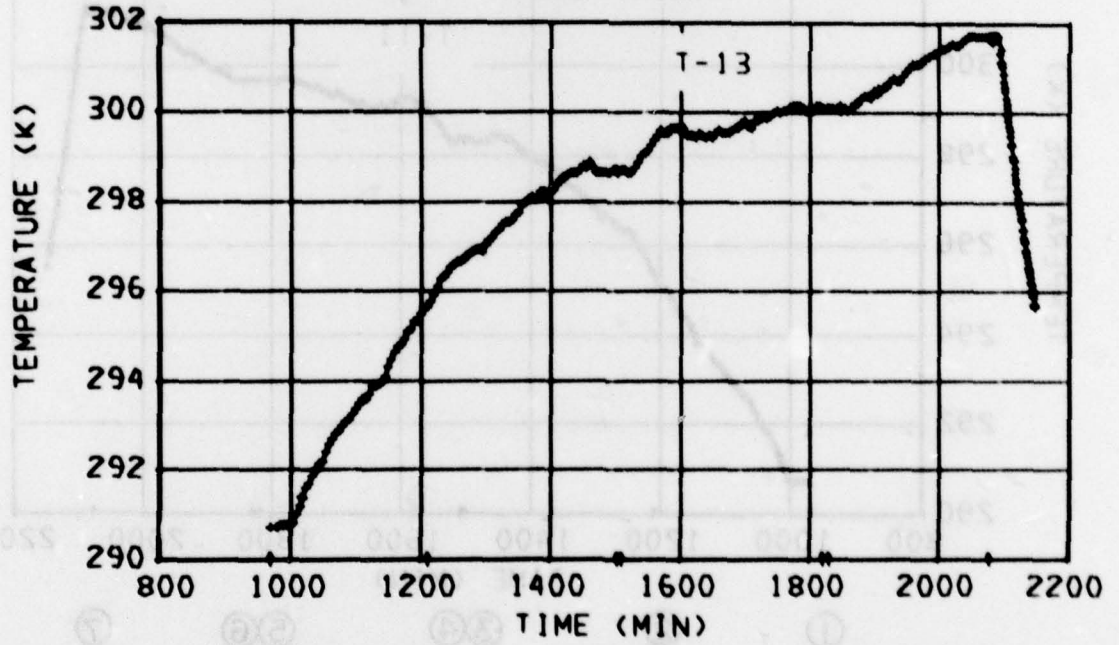

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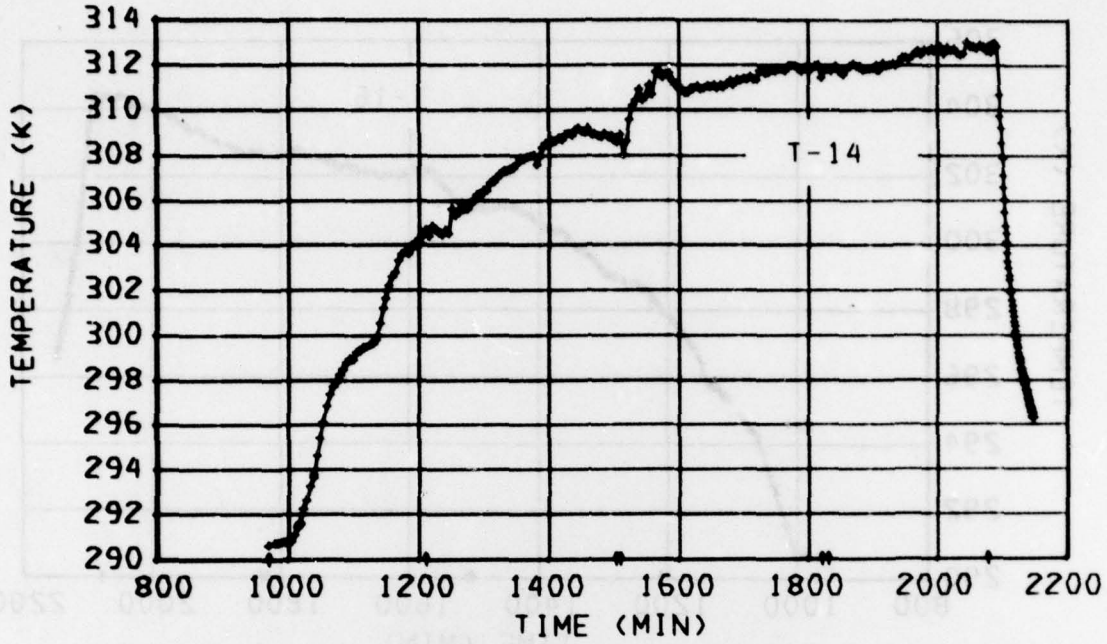

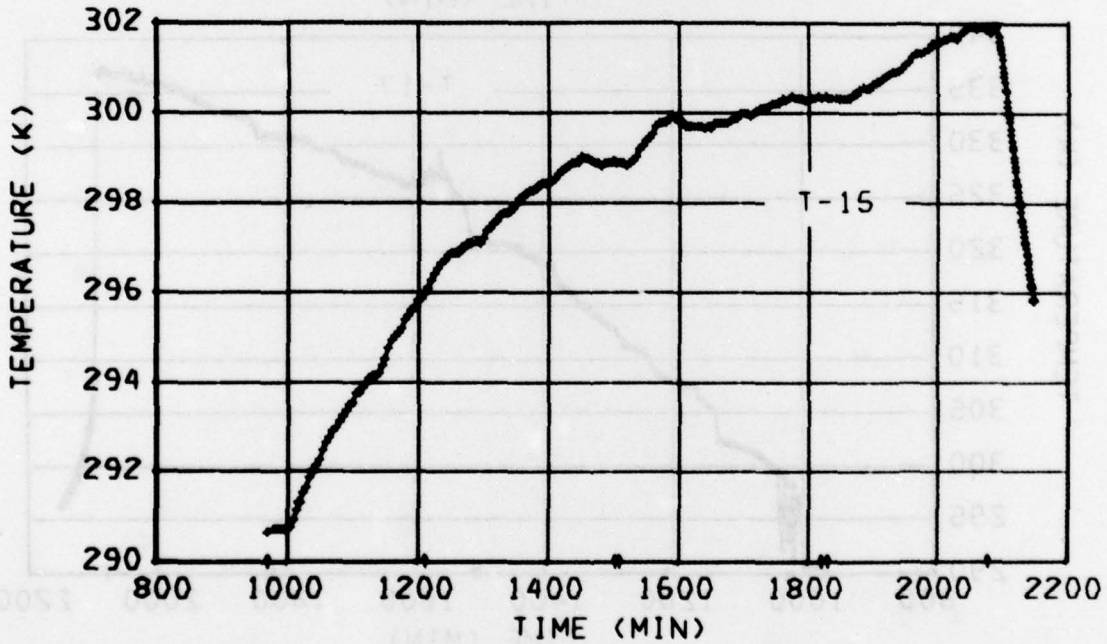

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DAS TIME OR REAL TIME (H)

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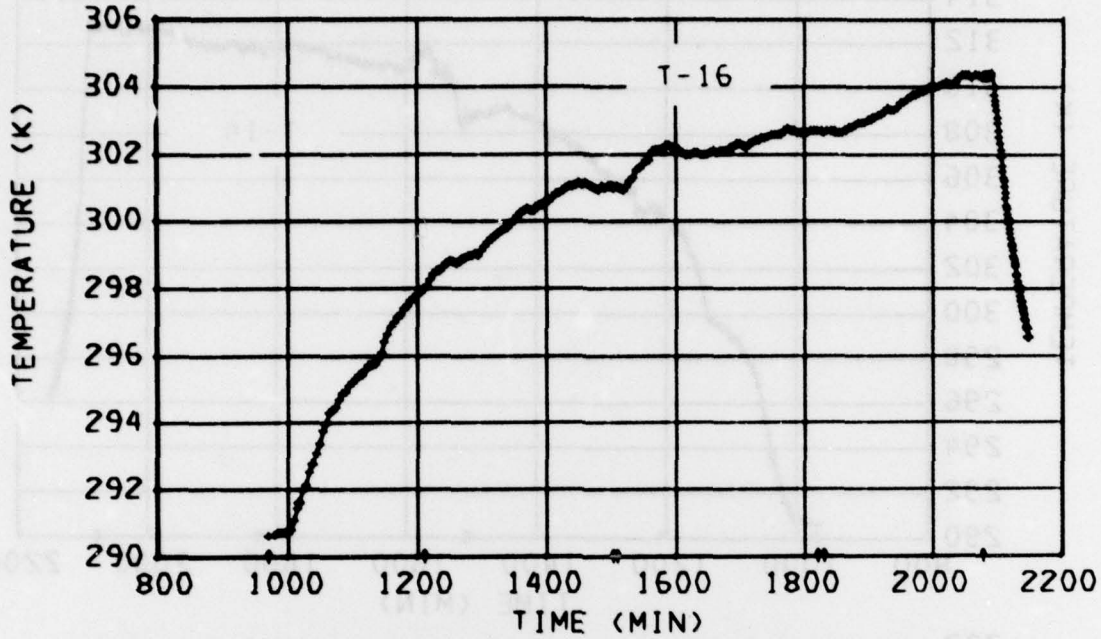

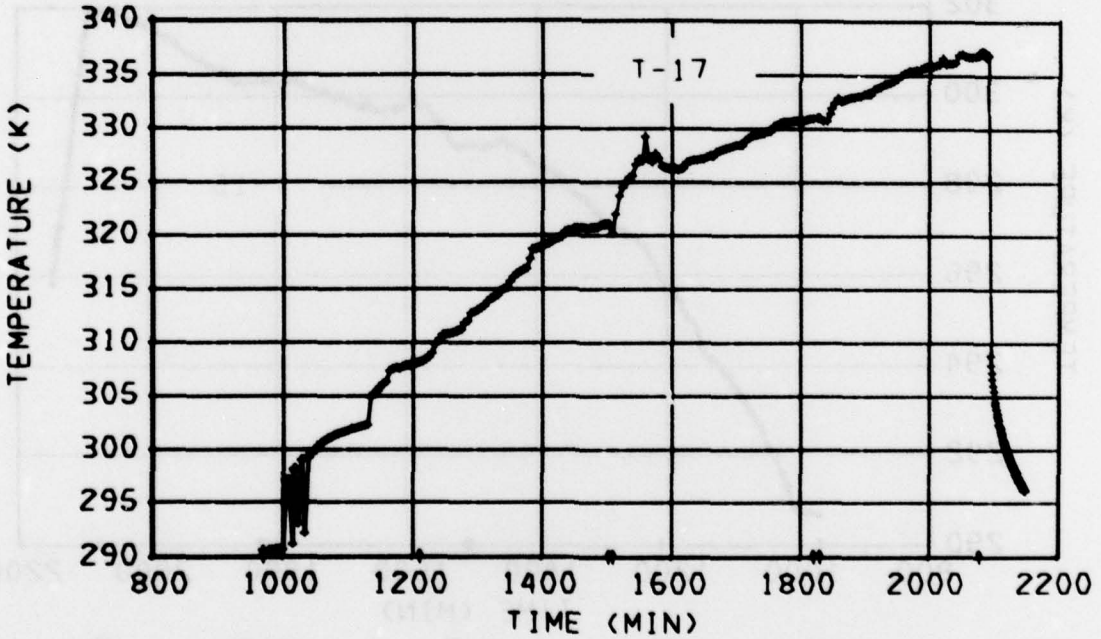

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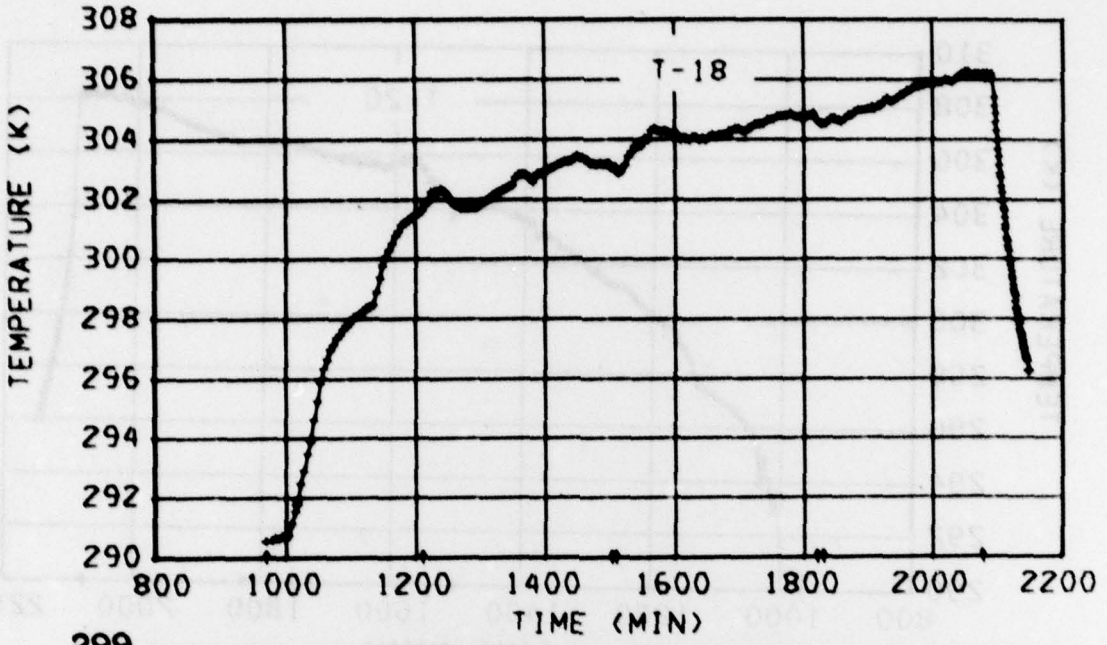

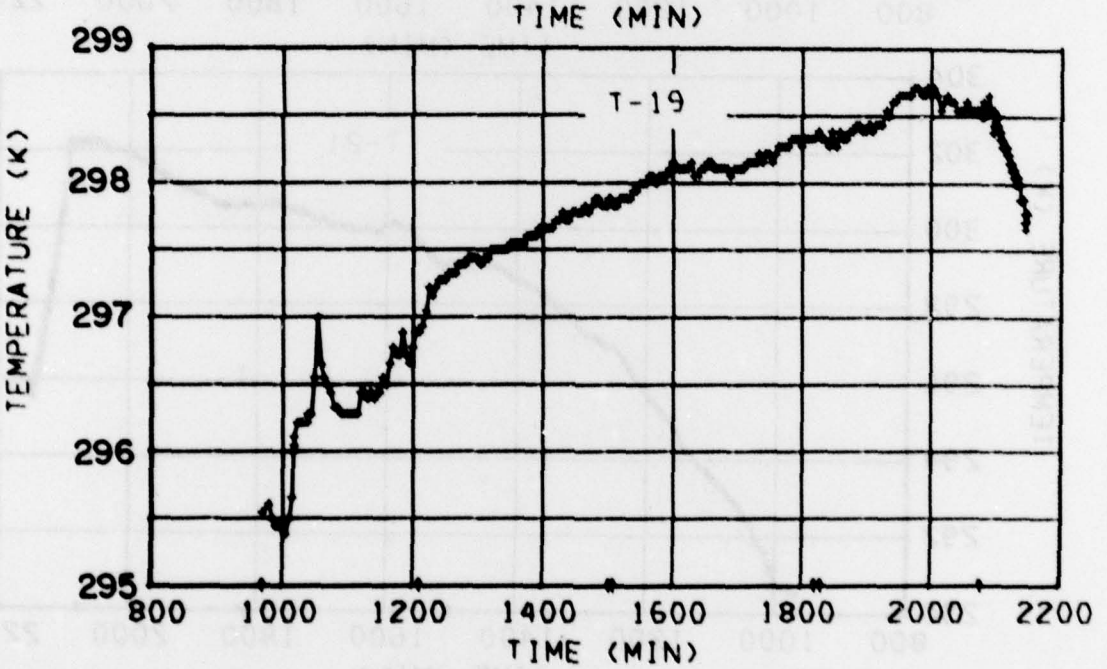

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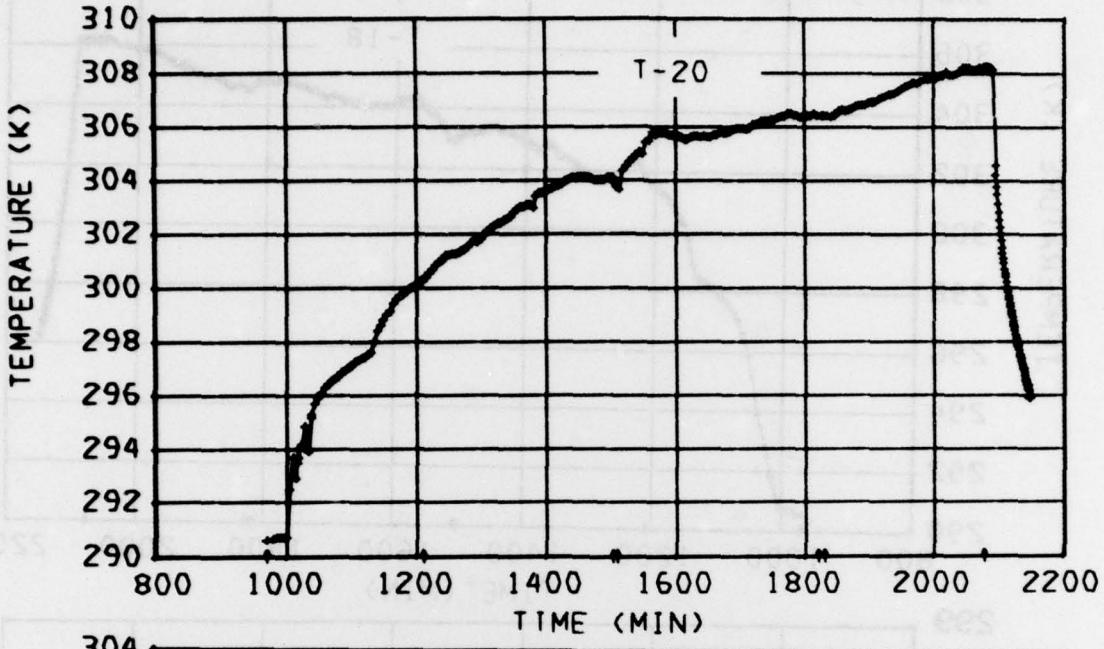

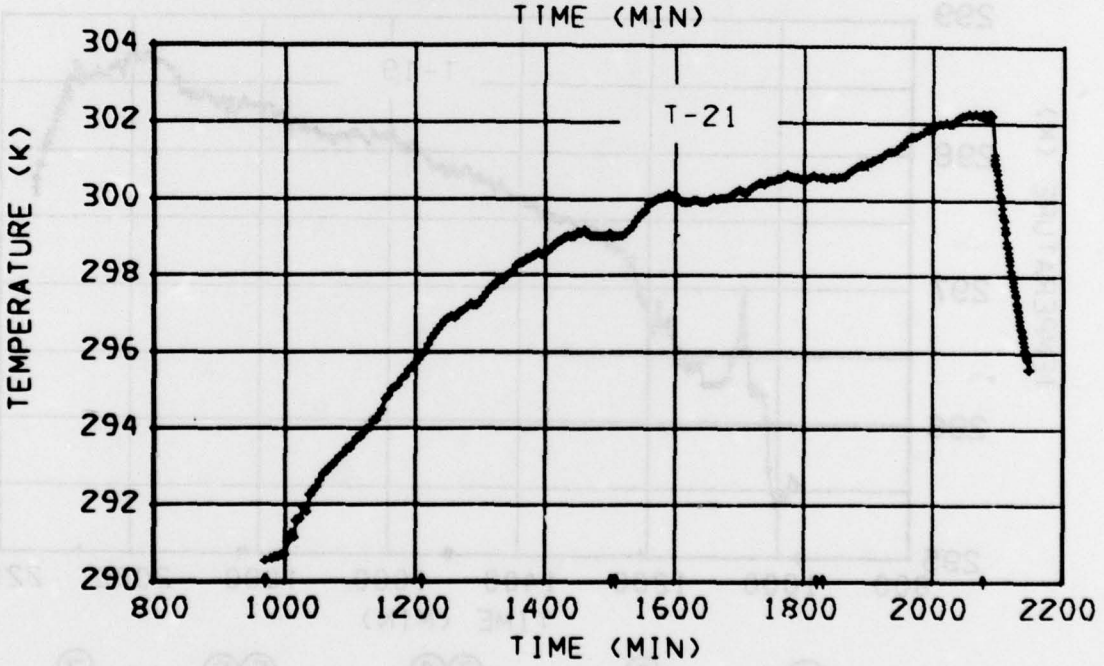

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DAS TIME OR REAL TIME (H)

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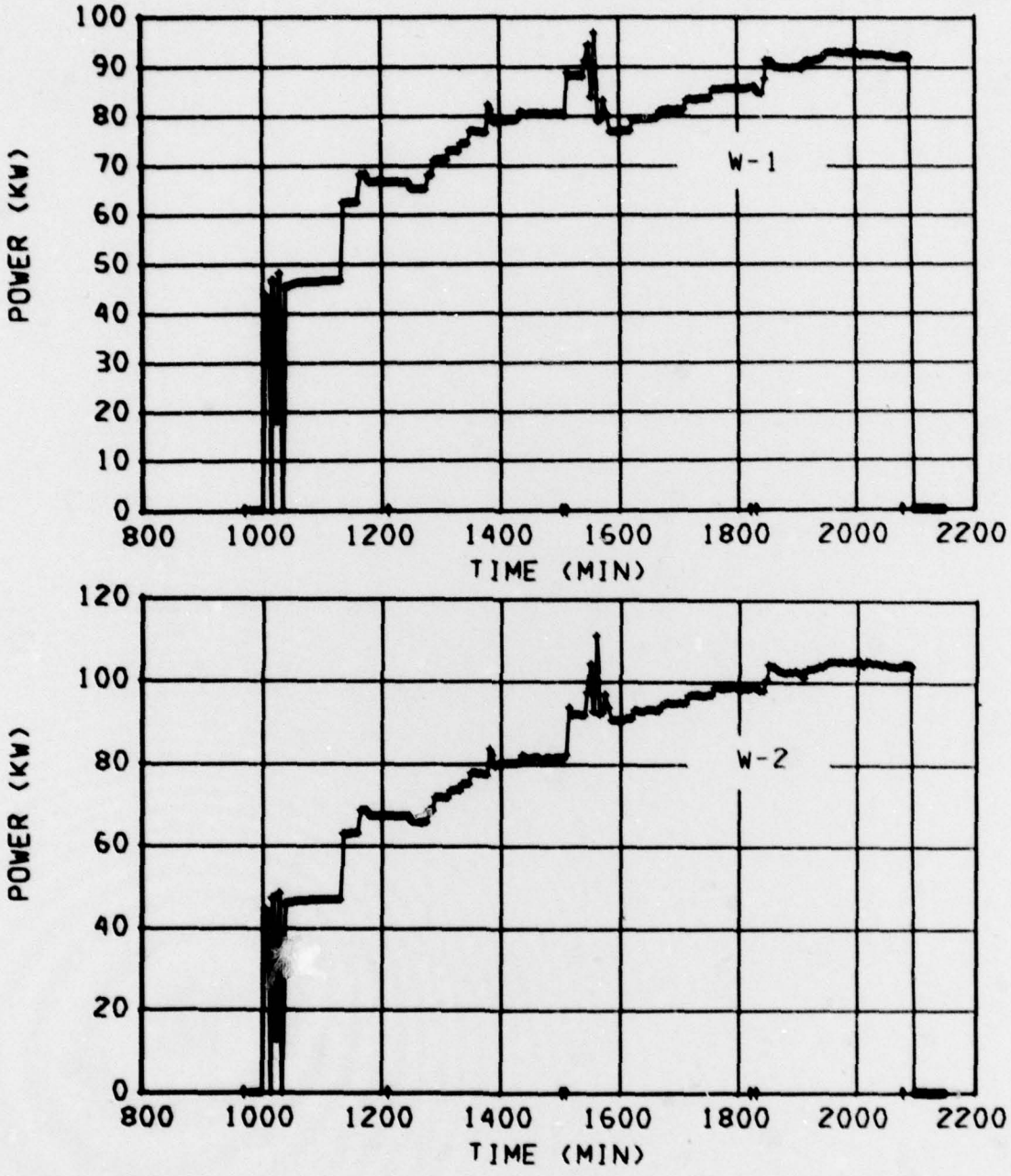

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